AMERICAN
DYESTUFF REPORTER

VOLUME XXIV AUGUST 12, 1935 NUMBER 16

Developments During 1934 in the

Chemistry of Silk and Silk Processing

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Chapter I

THE CHEMISTRY OF RAW SILK

Silk Cocoons and the Silkworm

REPORTS from Italian sources indicate that considerable attention is being devoted to a study of the silkworm in that country. In the previous review it was reported that a noticeable acceleration in the life cycle of the silkworm was produced by irradiating the silkworm eggs before hatching, and the larvae after hatching.

Several more recent studies deal with the feeding of the silkworms, and the effect upon the resultant silk of variations in their diet. One investigator determined that if the silkworms received insufficient food they produced a silk which contained a higher proportion of sericin and whose filaments were finer than normal. On the other hand there was no difference in the tyrosine and tryptophane content of silk from normally fed and from hungry worms.

The feeding of silkworms with mulberry leaves sterilized with silver fluoride was said to modify the relation of fibroin to sericin in the silk from these worms. However, this claim was disputed by another investigator.

The effect of moisture on the cocoons of the common silkworm (Bombyx mori) both during and after their spinning, has received further attention. Previously it was determined that lower yields of poorer quality silk were obtained if the moisture of the air in the spinning room exceeded 50%. As a result of further study it was concluded that the spinning rooms must be kept dry. A relative humidity far under 50% was highly advantageous. Also the temperature should be kept low, preferably well under 25° C.

A detailed study of the protein contained in the chrysalis of the domesticated silkworm (Bombyx mori), and the protein in the chrysalis of the Tussah silkworm, was made by three Japanese investigators. They heated the aqueous extract of freshly ground pupae in order to coagulate the protein which was then purified and analyzed. The table below shows the percentages of amino acids reported by these investigators and compares them with the amino acids obtained from the fibroin of Bombyx mori by Abderhalden in an earlier study which was reported in the previous review.

Three additional amino acids (choline, betaine and hydroxyproline) were isolated in small quantities from the chrysalis proteins. These amino acids were not mentioned in connection with the fibroin.

This article is the second in a series of reviews written exclusively for this journal. The first review appeared last year in this journal, starting with Issue No. 9. It summarized the progress which had been made up to the end of the year 1933 in connection with the fundamental chemistry of raw silk and the chemical processes which play a part in the transformation of raw silk into finished fabric. The present review continues the story through the year 1934.

As in the previous article, the subject is divided into the following five chapters:

I. The Chemistry of Raw Silk.
II. The Soaking of Silk.
III. The Degumming of Silk.
IV. The Weighting of Silk.
V. The Dyeing and Finishing of Silk.

Fibroin and the Proteins of the Chrysalis

Amino Acids	*Fibroin of Domestic Silkworms* %	*Chrysalis Domestic Silkworms* %	*Chrysalis Tussah Silkworms* %
Glycine	40.5	0.15	0.87
Alanine	25.0	3.79	2.52
Tyrosine	11.0	4.28	5.73
Leucine	2.5	6.04	5.68
Serine	1.8	...	Trace
Arginine	1.5	...	Small
Phenylalanine .	1.5	7.83	9.53
Proline	1.0	0.65	0.01
Lysine	0.85	...	1.00
Histidine	0.75	...	2.84
Aspartic Acid..	trace	2.60	0.47
Glutaminic Acid	trace	1.70	5.62

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Silk Reeling

A German patent dealing with an improved method for reeling silk was issued in 1934. This patent supplemented a previous American patent on the same subject. It claimed that better reeling resulted from pretreating the cocoons, first with a dilute solution of ammonia or an amine, and then with a dilute solution of a caustic alkali. The ammonia or amine bath preferably contains a salt, such as sodium acetate or ammonium acetate.

The Chemistry of Sericin

During 1934 there was another addition to the sericin family. Sericin A and Sericin B, a complete description of which was given in last year's review, were joined by Sericin C according to Mosher. However, there appears to be some question as to whether this new sericin is a legitimate member of the family. Mosher states that Sericin C comprises the innermost layer of the silk gum fraction, also that it is insoluble in all common solvents, almost completely resistant to enzymes, and possesses but slight ability to emulsify oils and fats. These properties set it apart from Sericins A and B which are chemically closely allied to each other.

Mosher's work was to some extent corroborated by a Japanese investigator, but the latter drew a somewhat different conclusion as the result of his studies. He claimed that, during the degumming of silk, after the last traces of sericin were removed there still remained an intermediate product which was neither sericin nor fibroin. It was necessary to extend the degumming over a period of 2 to 3 hours in order to remove this intermediate product as well as the last traces of sericin. During this operation a proper concentration of alkali was essential in order to prevent decomposition of the surface of the fibroin.

Further studies with Sericin A and Sericin B indicated that the former had more resistance to oxidation with potassium permanganate and sulfuric acid than the latter. The resistance of the sericin to oxidation proved to be parallel with the reelability of the cocoon from which the sericin was prepared.

The Coloring Matter of Silk

The pigments in Italian and Japanese silk may be extracted with acetone and with ethyl alcohol. The yellow pigments consist primarily of carotenoids as may be shown by their behavior with reagents such as antimony trichloride in chloroform, ferric chloride, sulfuric acid, carbon disulfide, etc. The green pigments, on the other hand, give typical flavone reactions with ferric chloride, sulfuric acid and basic lead acetate.

The Chemistry of Fibroin

Studies on the hydrolysis of silk fibroin have been a fruitful field for investigation since 1922 when Abderhalden announced a list of amino acids which were isolated from the product of this hydrolysis.

During the past year a Japanese investigator reported the results of an extended study on the partial hydrolysis of fibroin. It was found that fibroin could be kept in concentrated sulfuric acid for many weeks without any marked increase in the free amino groups. With sulfuric acid of 70% strength the hydrolysis was much more rapid.

When fibroin was treated in an autoclave at 170° C. with 0.15-0.175 N sulfuric acid, the increase in amino groups was very large at first, but gradually diminished and finally ceased entirely at the end of 7½ hours. At this time 42-43% of the total nitrogen was in the form of free amino groups and the biuret reaction had disappeared. With 0.1 N sulfuric acid the biuret reaction persisted even after 12½ hours in the autoclave. With 0.2 N sulfuric acid the hydrolysis was very rapid and the amino nitrogen continued to increase even after the biuret reaction disappeared. With dilute sodium hydroxide the hydrolysis was always more rapid than with acid, and the increase in amino nitrogen was continuous with all concentrations of the alkali.

Fractionation of the product resulting from the hydrolysis with 0.15-0.175 N sulfuric acid, produced an anhydride of glycine and tyrosine upon evaporation. Anhydrides of glycine with alanine and with tyrosine were obtained from the acetic ester extract.

From these and other studies it was concluded that the primary products of hydrolysis were polypeptides which were either changed partly to diketopiperazines or hydrolized to amino acids. In the fibroin molecule glycine must be in juxtaposition to alanine and to tyrosine, alanine is probably not next to tyrosine because no anhydride of these two was isolated.

Solutions of Fibroin

Fibroin may be put into solution with the assistance of the following agents: (1) alkaline solutions of copper oxide, (2) alkaline solutions of other metallic hydroxides, (3) concentrated solutions of sodium or calcium thiocyanate, (4) concentrated solutions of other salts (such as sodium iodide, calcium chloride and magnesium nitrate), (5) strong phosphoric or sulfuric acid at low temperatures, and (6) liquid ammonia at about 77° C.

An Italian investigator reports that a solution of silk is an irreversible colloid. It is coagulated by the gentlest rubbing and by passing through a capillary. It is precipitated by dilute acids and coagulated by concentrated acids. A solution of caustic soda (0.001 N) retards coagulation. Solutions of electrolytes must be highly concentrated to cause coagulation. The coagulum is inactive when formed spontaneously, but quite active when formed mechanically.

The mechanism of the solution of fibroin in alkaline solutions of copper oxide received further attention in the year 1934. It was found that the following compounds may be used to accelerate the solution of fibroin in mixtures of copper hydroxide with alkali:—

(1) Substances which will dissolve copper hydroxide without the aid of caustic alkali.

(A) Ammonia, ethylene diamine, etc.
(B) Di- and tri-ethanolamine, biguanide, etc.

(2) Substances which will dissolve copper hydroxide with the aid of caustic alkali.

(A) Amino acids—such as glycocoll, alanine, etc.
(B) Polyhydroxy compounds—such as glycerol, glucose, etc.

Some of the above catalytic agents are very effective. As little as 0.04 gram of ethylene diamine to 100 c.c. of the alkaline copper hydroxide solution will reduced the time required to dissolve the fibroin from 40 hours to only a few hours.

When fibroin is dissolved in copper oxide-ethylene diamine two definite compounds are formed in which the ratios of fibroin to copper to amine are 1:1:1 and 1:2:2, respectively. This conclusion has been substantiated by conductometric titrations. When sulfuric acid is added to the system copper-ethylene diamine-hydroxyl there is an initial fall in conductivity. The lowest point is reached with the neutralization of the hydroxyl groups. From this point the conductivity increases until there is a break in the curve at the point of neutralization of the total basicity of the copper and the ethylene diamine. For the compound (fibroin Cu) ethylene diamine there is one break in the curve; and for the compound [fibroin Cu] [Cu (ethylene diamine)$_2$] there are two breaks in the curve.

When ethylene diamine is simply used as a catalyst to promote the solution of fibroin in copper hydroxide-caustic alkali mixtures, it is believed that the fibroin reacts first with the copper-amine base and then further with the alkali to form a fibroin-copper-alkali compound. In this compound the ratio of fibroin to copper to alkali is 10:4:10 irrespective of whether the alkali is sodium hydroxide, potassium hydroxide or lithium hydroxide.

Commercial Applications of Fibroin Solutions

A Japanese patent deals with the regeneration of silk thread from solutions of fibroin in salts such as calcium nitrate, calcium thiocyanate or sodium thiocyanate. The solutions are forced through small orifices into coagulating baths consisting of solutions of sodium sulfate and zinc sulfate acidified with sulfuric acid.

The use of anhydrous ammonia as a solvent for fibroin has also received further attention in the patent literature. It is claimed that the solution is more easily effected if the degummed silk is first immersed in a boiling solution containing 1.5 to 3.0% of an aliphatic organic acid such as acetic acid, then dried while still wet with the acid, and finally entered in the liquid ammonia at 77° C.

A study has been made of the effect of viscosity in connection with the coagulation of the fibroin solution. It was found that when the difference between the viscosity of the fibroin solution and that of the coagulating solution was larger, the film formation of the regenerated fibroin was made comparatively easier.

Solutions of fibroin have also been recommended for the purpose of giving a silk-like finish to cotton or other vegetable fibers. The cotton is first treated with a solution of a caustic alkali and then with a colloidal solution of silk.

Inorganic swelling agents which are capable of dissolving silk may be used in a mild form to increase the transparency, change the luster and alter the dyeing properties of the silk. The type of swelling agent used, its concentration and the temperature are important factors, but the length of the treatment is the most important of all. Mild swelling agents tend to equalize the thread size while stronger agents tend to increase the transparency. Solutions suitable for this purpose are zinc chloride (65° Be.), sulfuric acid (54° Be.), and sodium hydroxide (Sp. gr. 1.21). The temperature may be varied from 5 to 50° C. and the time from 5 to 60 seconds.

Additional Properties of Silk

A method has been developed for determining the electrical conductance of silk, as well as other textile fibers. The electrical conductance of degummed silk is less than that of raw silk. The conductance varies considerably with the method of degumming: for example, silk degummed with soap or with a caustic soda solution has a higher conductance than silk degummed with an enzyme such as pancreatin. Silk treated with water at a temperature above 121° C. and a pressure above 2 atmospheres is a better conductor than silk scoured with soap. In treating degummed silk with solutions of caustic soda, the electrical conductance increases with the concentration of the alkali.

A study has been made of the effect on silk of boiling it in solutions of ammoniumchloride. It was found that only hydrogen chloride is adsorbed by the silk and ammonia distills off. The amount of adsorbed acid is increased with an increase in the concentration of the ammonium chloride in the solution. The saturation limit for the silk is 200% of ammonium chloride. This is higher than theoretical, which may be explained by a partial hydrolysis of some of the acid amide groups in the silk molecule.

Chapter II

THE SOAKING OF SILK

Sulfonated Oil Emulsions

As a result of an investigation sponsored by the Textile Foundation much additional data has been obtained with reference to the absorption of oil by silk from emulsions in which sulfonated oils are used as the emulsifying agents. In making a critical study of the soaking of silk, the following factors must be taken into account:

(1) The ratio of volume of oil emulsion to weight of raw silk.
(2) The effect of temperature upon oil take-up.
(3) The effect of hydrogen ion concentration both in the initial and final stages.
(4) The effect of various types of oils, with special regard to unsaturated fatty acids, unsaponifiable matter, etc.

(5) The effect of various emulsifying agents upon the resultant oil take-up.
(6) The effect of free and combined oils upon the character of the resultant silk.
(7) The effect of time upon oil take-up.

In determining the amount of oil taken up by the silk in the soaking process a distinction is made between the free oil and the combined oil. The free oil is that portion of absorbed oil which can be removed from the silk by a simple extraction with ether. The combined oil can only be determined by first hydrolizing the silk with a 20% solution of hydrochloric acid and then extracting the residue with carbon tetrachloride.

When raw silk is soaked in oil-water emulsions the take-up of both free and combined oil increases with an increase in the hydrogen ion concentration of the emulsion in the range between pH 4 and pH 9. The changes in pH seem to have a greater effect upon the take-up of the free oil than of the combined oil. If increasing percentages of raw oil are added to the sulfonated oil emulsion the take-up of free oil is increased but the take-up of combined oil remains practically the same. In general, when the pH of the initial emulsion is below 6 there will be an increase in pH during the soaking process, but with an initial pH of over 6 there will be a decrease during the soaking.

The take-up of both free oil and combined oil increases with the temperature of the emulsion in the range between 10° C. and 40° C. However, changes in temperature do not have nearly as great effect upon the oil take-up as changes in pH.

An increase in the concentration of oil in the bath causes a more rapid and increased amount of oil to be taken up, as might be expected. In practically all cases the take-up of oil increases quite rapidly in the early stages of the silk immersion and then remains constant for quite a period, after which the take-up increases again. It is believed that the second increase in the take-up after an extended period of time is the direct result of bacterial rather than chemical action. The silk fiber is attacked by the bacteria and the subsequent decomposition affects the oil take-up. That such a theory holds is evidenced by the development of a distinct odor at this stage of the process.

Other Emulsifying and Penetrating Agents

The following classes of agents have been shown to have emulsifying or penetrating properties which would make them of interest for the soaking of silk:

1. The sodium salts of alkylated naphthalene sulfonic acids.
2. The sulfonated abietenes.
3. The sodium salts of sulfonated esters of fatty acids or acid amides.
4. The sodium salts of fatty alcohol sulfates.
5. Solvents (such as pine oil, cresol, tetralin, hexalin, etc.)

A number of methods have been proposed for determining comparative efficiency of various wetting agents in the penetration of textile fibers. The methods may be divided into three groups as follows:—

1. Measurements of the forces acting at the surface of the solutions, i.e., surface tension measurements or comparisons of the capillary rises in threads dipping into the solutions.

2. Determinations of the time required for fibers or fabrics to wet-out and sink in the solutions.

3. Measurements of the amounts of the solutions absorbed during a given period of time by the fibers or fabrics.

A recent study indicated that the last method was most suitable for evaluating the penetration into cotton yarn. It is believed that this method would be equally satisfactory for determining the value of these agents in the soaking of silk. In this method a skein of known weight is soaked in the solution at a definite temperature for a given time, and then centrifuged under standard conditions and weighted.

A rapid method for determining the degree of exhaustion of soaking solutions has been developed with the aid of the Babcock milk tester. With this instrument it is possible to measure directly in a very short time the amount of oil in a definite quantity of the soaking solution. Examinations of the soaking bath may be made before, during and after the soaking.

The Soaking of Hosiery Yarns

The throwsters of hosiery yarns have in recent years developed methods of soaking which will deliver to the hosiery mills silk requiring no back-winding over wetting-out agents and very little or no conditioning before knitting. This has been accomplished by raising the pH of the soaking bath. Formerly the pH was about 8.3 but it has been raised to a point slightly below the pH at which the sericin would be solubilized. A pH of 9.3 has been found to be quite satisfactory. If the pH is raised to about 10.0 the gum will show a tendency to strip from the silk during the knitting process.

Chapter III

THE DEGUMMING OF SILK

A recent detailed investigation of silk degumming has led to the formulation of a new theory on the mechanism of this operation, and to the specification of two new control factors as follows:

1. The binding weight of the sericin; i.e., the grams of sericin combined with one gram equivalent of alkali in the silk gum solution. This is a measure of the degradation of the sericin during degumming.
2. The degumming coefficient i.e., the proportion of total sericin removed from the silk in a given time. This is a measure of the effectiveness of the various degumming agents.

The Binding Weight of the Sericin

A lowering of the binding weight indicates an increased degradation of the sericin. The binding weight of sericin varies from 2100 when it is peptized by distilled water

down to 140 when it is dissolved by tenth normal sodium hydroxide. The binding weight decreases with increasing values of pH in the case of each degumming agent, but in general it is controlled by the ratio of alkali to sericin initially present, rather than by the nature of the alkali or by pH values.

The Degumming Coefficient

In the case of soap and of other alkalies, a higher degumming coefficient indicates a greater effectiveness in the removal of the sericin from the silk. The degumming coefficients of alkalies (other than soap) are nearly independent of concentration and are specific for each reagent. The degumming coefficient of a soap solution varies with its concentration. The most efficient concentration is 0.053 Normal and the efficiency decreases both above and below this concentration.

On the basis of equal weight in pounds the boil-off effectiveness of the alkalies is in the following order:

1. Sodium hydroxide.
2. Sodium metasilicate ($Si\ O_2/Na_2\ O = 1.0$).
3. Sodium carbonate.
4. Sodium silicate S. ($Si\ O_2/Na_2\ O = 2.62$).
5. Trisodium phosphate.
6. Sodium borate.
7. Sodium bicarbonate.

Soap covers the range of the milder alkalies up to and including trisodium phosphate.

The degumming coefficient is also controlled by the temperature. For example, with a 0.03 Normal soap solution the degumming coefficient increases from 33 to 100 as the temperature increases from 180 to 200° F.

The Degumming of Silk Hosiery

In hosiery degumming the finish is said to be dependent upon the amount of residual sericin left on the hose after the boil-off. When soap is used as the degumming agent it is possible to control the residual gum content on the silk by suitable adjustments of the time, the temperature and the soap concentration.

In the case of particularly hard lots of silk the soap solution may be strengthened by the addition of certain alkalies. The following alkalies are used quite extensively for this purpose: sodium carbonate, sodium borate, trisodium phosphate, sodium silicate and sodium sesquicarbonate (a mixture of the carbonate and the bicarbonate). The last named is thought by many hosiery dyers to be the most suitable.

One investigator states that sulfonated oil is more suitable than soap when the one-bath method for degumming and dyeing silk hose is used. The advantages claimed for the oil are: (1) better finish and feel, (2) more level dyeing, (3) less likely reduction of dyes, (4) longer runs of the batch without harshening effect, (5) more accurate formulas, (6) easier and quicker emulsification of the soaking oils, and (7) greater economy.

Degumming with Enzymes

A recent patent proposes a new and novel method for degumming of silk with enzymes. The raw silk is impregnated with a protease preparation in the presence of a suitable penetrating agent, then dried without the elimination of an appreciable amount of gum. The silk is subsequently degummed by immersion in hot water.

Chapter IV

THE WEIGHTING OF SILK

Modifications of the Regular Tin Weighting Procedure

The patent literature continues to propose new modifications of the usual weighting procedure consisting of alternate immersions in solutions of stannic chloride and disodium phosphate and a final treatment with sodium silicate.

Silk may be weighted by treating it in a bath at 40-50° C. containing tetrachloride of tin, magnesium chloride and monsodium phosphate, whizzing, treating in a cold solution of phosphoric acid, whizzing, treating at 60° C. in monosodium phosphate, washing and then treating in a silicate bath at 55° C. In a second weighting cycle the weighting solution may contain aluminum acetate or aluminum formate and acetates of lead and magnesium, and the fixation may be again completed with silicate.

The weighting bath may be a colloidal solution of basic zinc—tin chloride prepared by adding zinc carbonate to a solution of tin tetrachloride. Sodium tungstate or sodium molybdate may be used in the fixing bath in place of sodium phosphate, or salts (such as sodium chloride and sodium sulfate) may be added to the phosphate solution to hasten its action.

In another modification, after alternate treatments with tin and with phosphate, it is proposed to wash the silk with dilute acetic acid and treat it in a solution of zinc acetate, followed by washing, then disodium phosphate and finally sodium silicate.

The Clavel weighting process was described in the previous review (issue of June 4th, 1934) and need not be repeated here. In a recent patent Clavel proposes to substitute chloroacetic acid as a swelling agent in place of formic or hydrochloric acids. A preliminary treatment in a solution of this acid makes the silk more receptive to the tin solution.

The Analysis of Weighted Silk

A detailed procedure for the analysis of tin weighted silk has recently been described. The sample is digested with concentrated nitric and sulfuric acids, treated dropwise with hydrochloric and hydrobromic acids at 140-180° C., and the tin is distilled off. It is either precipitated with hydrogen sulfide, or determined volumetrically after neutralizing the excess of acid in the distillate with ammonia. Silica in the residue is collected on a filter. The phosphate is determined by precipitation with 8-hydroxyquinoline and a soluble molybdate.

Within certain limits the degree of weighting may be determined by the swelling of the silk in a cuprammonium hydroxide solution. The swelling power decreases in proportion as the amount of weighting increases.

Chapter V
THE DYEING AND FINISHING OF SILK

Mordanting

A study has been made of the absorption of chromium salts by natural silk. The amount of chromium oxide fixed by silk increases with the concentration of the bath up to about 1% strength. Beyond that point there is no further increase in the absorption of chromium oxide no matter how strong the solution may be. The fixation of the chromium oxide on the silk also increases with the temperature of the bath up to boiling, with the time of immersion up to 30 minutes, and with the alkalinity of the bath. The addition of mineral acids to the chromium bath decreased the absorption but the addition of neutral salts had very little effect.

Dyeing and Printing

One of the few papers on silk dyeing during the year 1934, appeared in the Russian Journal of Applied Chemistry. This article dealt with the dyeing of silk with substantive dyes in the presence of ammonium chloride. By distillation of the dyed silk it was found that the amount of ammonia which distilled over was equivalent to the amount of dye adsorbed. Thus it was concluded that the silk adsorbed only the anions. By raising the amounts of ammonium chloride and of dye in the bath, the absorption of dye by the silk was increased to a saturation limit of 0.0002 to 0.00023 gram equivalents of dye per gram of air-dry fiber. The maximum absorption was reached only with large amounts of ammonium chloride in the bath and prolonged boiling.

Further emphasis was placed on fast color dyeing and printing. In connection with the dyeing of vat colors on silk, emphasis was placed on the lack of affinity of the silk for this class of dyes and it was recommended that the silk be thoroughly scoured prior to dyeing in order that the dyed material might be as free from crocking as possible. Additional patents dealt with the printing of silk with chrome colors. The methods were similar to those described in the previous review, the principal feature being the addition of urea, an aliphatic polyalcohol, or other designated compounds to the printing paste.

Finishing

A special decorative finish may be produced on silk by a method of resist printing. A mixture of one part of printing gum to two parts of gum Acacia is printed as a resist on the silk and the fabric is then treated in formic acid to straighten and compact the exposed threads. The acid and the resist are then removed by rinsing.

As mentioned earlier in this article under the chemistry of fibroin, silk may be given a transparent effect by finishing it in a solution of some swelling agent, such as zinc chloride, sulfuric acid or sodium hydroxide. The swelling bath should be cold and it should contain a protective agent, such as alcohol, pyridine or an ammonium salt. The treatment should last about 30 seconds.

Treatment of Waste Liquors

It is recommended that the printing wastes and boil-off liquors be separated from the other silk wastes. By so doing, the remaining liquors can be blended and economically treated with a coagulant to remove the color and to make the effluent satisfactory for the supporting of fish and plant life. In general a mixture of 4 pounds of copperas and 4 pounds of lime is sufficient for the coagulation of 1,000 gallons of silk waste liquors. The amount of coagulant required may be substantially reduced if the waste liquors are given a preliminary sedimentation and screening.

The Testing of Finished Silk

It is well known that silk gradually loses its tensile strength upon exposure to sunlight. Recent studies have shown that this decrease in strength is accompanied by an increase in the ammonia nitrogen content. This is additional evidence that the decomposition of the silk is caused by atmospheric oxidation in the presence of light. Further confirmation of this theory was obtained by exposing skeins of silk in a vacuum and in atmospheres of moist and dry hydrogen and oxygen to north skylight for 4 months. The skeins in the vacuum and in the hydrogen lost no strength during this period, while the skeins in the dry oxygen lost about 3%, and those in the moist oxygen about 10% of their strength.

Samples of silk fabric treated with a tenth normal solution of sodium hydroxide showed smaller losses in strength under the action of light and air than the untreated fabric, while those treated with a tenth normal solution of sulfuric acid showed greater losses. The maximum stability toward sunlight was obtained by treating the silk in a sodium carbonate solution of 1% or less in concentration.

New methods have been developed for determining the comparative shrinkage of various silk fabrics and also the comparative slippage. These methods will take their place with the tests which have already been recommended for determining the fastness of dyed silk to washing and to light.

The measuring and recording of small color differences in dyed silks is made considerably easier by improvements in the use of the recording spectrophotometer. A recent article dealt with the detection of small color differences on wool, and the technique used in obtaining the data would be equally applicable to dyed silk. By this method of color measurement it was possible to detect differences in color where the difference in the amount of dye used was only 0.0001% based on the weight of the silk.

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● DU PONT EXHIBIT

Depicting the progress of chemical industry and dramatizing the part that the "invisible science" plays in every day life, 21 manufacturing divisions of the E. I. du Pont de Nemours & Co., Inc., will show their products and indicate the processes by which they are produced at the Eastern States Exposition in Springfield, Mass., from Sept. 15 to 21 inclusive.

The exhibit, "The March of Chemistry," will occupy 356 running feet in the Exposition's three-acre Industrial Arts Building and will be the largest single display ever presented at an annual exhibition. It will be of educational nature and will cover a wide range of chemical products, including several spectacular new developments of du Pont research laboratories, showing how they are made, how they are used and how they serve, often in an unseen way, the ultimate consumer.

Emphasis will be laid on how the du Pont products enter into the finished articles, contributing to their beauty, value and utility. Each booth will be supplemented by displays of modern merchandise. In addition to the products which du Pont itself markets in finished form, there will be hundreds of other articles manufactured by other companies, made wholly or in part from du Pont materials. Thus the exhibit as a whole will give a graphic picture of the many chemical operations, unseen by the final purchaser, required in producing quality merchandise.

The exhibit will be under the direction of William R. Hart and R. A. Applegate, director and assistant director of du Pont advertising.

● INCO

The summer issue of *Inco*, containing information concerning uses of products of the International Nickel Company, has recently been issued. Copies of this publication may be obtained on request.

Factors Affecting

Roll Treatment

of Textile Fabrics

By G. L. ATKINSON

THE fact that textile fabrics are treated in roll form in most preparing, dyeing and finishing operations, and in the most important of these operations, is a fact that if it has been realized, has not yet been appreciated to the full.

That this matter of roll treatment is more than important, and fully worthy of further comment, may be judged by the fact that many faults are caused by neglect to appreciate this factor to the full. These faults are caused by the rollers in use and in addition the production and other costs together with efficiency are affected to a degree as yet hardly realized by this factor.

The importance of roll treatment of fabrics has gained in significance since the introduction of piece work generally in the textile industry. Apart, however, from any question of piece work, or any other working scheme, it is seldom now that pieces are treated separately or individually in any preparing or finishing process, that is to say, the object today is to enable the work to move forward as continuously as possible.

The goods are treated in roll form, in the grey room, the conveying of the goods from the grey room to the preparing departments, on the crabbing machine, the steaming process, in some dyeing processes, some hydro-extracting methods, and in decatizing during finishing, a particularly important finishing operation.

There are many ways of conveying the grey pieces to the preparing departments, these generally depending upon the situation of the two departments. The goods are generally conveyed by hand labor (a very crude and costly method in addition to being very slow), in carts or trucks, or in the case of the two departments being on different floors of the same mill, by lift. With all these methods there is always the risk of the pieces being damaged, while costs are high and the production is not very great.

There is one method however that is applicable to the conveying of the goods, no matter where these two departments may be situated, and what is more important this system is applicable to any type of textile fabric, without the least alteration. Here the goods are treated in roll form and are beamed. The cost of the apparatus is not great, the goods being run from the grey room, wherever it may be situated to a power drive, which includes a loose roller on which the goods are wound. The part of the apparatus in the grey room is equipped with tension rails, the tension at which the goods are wound upon the roller then being adjustable. Any number of pieces may be sewn together in the grey room for crabbing, scouring or milling, all that is necessary being that each group of goods for individual treatment be separated from the next batch by a long cotton crabbing wrapper. The batches can be easily detached from each other when they reach the preparing department. A feature of the goods being beamed is that as the goods are wound upon the roller in the power drive, the progress of the goods becomes quicker and quicker as the roll of pieces and wrappers becomes bigger. This is particularly important when production is of importance. In addition the goods are wound upon the roller at open width, which is seldom the case when the goods are conveyed either by hand, cart, or lift. The tension upon the pieces also throughout the operation is constant, an important factor in the crabbing of the pieces; the tension may also be changed without trouble to suit the individual requirements of certain types of piece goods. Finally there is little or no risk of the goods being damaged or stained and when finally wound upon the roller in the power drive the whole of the pieces are effectually covered by the outside wrapper. This acts as a cover from dust, dirt, and oil in the preparing departments.

There is also the question of labor changes in any textile concern and with this system in operation it will be found that only three men are necessary for operating the system, one being in the greyroom, the other two being employed at the power drive end of the machine, and for lifting out the full rolls and replacing empty rollers in the power drive.

This takes us to the next operation where the goods are treated in roll form, namely crabbing. Here the goods are wound upon rollers, in one or more troughs, in boiling liquor for the correct setting of the cloth. In the crabbing operation both tension and pressure are not only important, but are vital. Here the benefits of the goods being beamed when conveyed from the greyroom are fully realized. In many concerns even today, the goods are run from a heap on the floor at the back of the crabbing machine or out of a cart or truck. These methods are responsible for many of the faults that are caused in fabrics during the crabbing operation, in fact uneven tension is the root cause of much unsatisfactory crabbing.

It is essential that during the running of the cloth into the crab bowls that the tension shall be constant. This is achieved without further trouble if the goods are beamed prior to the crabbing operation.

The further instance of roll treatment is when the goods are wound upon the crabbing roller in the troughs of the crab. Here both tension and pressure play an important part in the crabbing of the goods. Obviously there is much greater pressure exerted upon the inner layers of the roll on the crabbing roller than on the outer layers of the roll. In the case of poplin and repp cloths, this nearly always leads to watering or embossing taking place. To take the example of much easier cloths from a crabbing point of view, namely serges, here the fabric is often cross-twilled, this being entirely due to the pressure and tension when the goods are wound in roll form upon the crabbing roller. Many state that effects caused by this means can in certain instances be rectified when the goods are run into the second crab bowl, for in this instance, the order of the goods are reversed upon the roller and the former inner layers are now on the outside of the roll. This certainly levels up the effects so far as the setting of the fabric is concerned but it does not affect any faults that are caused in the first bowl of the machine.

Similarly many concerns are of the opinion that the number of pieces that can be crabbed together is only limited by the capacity of the crab bowls. This may vary with different machines; in some crab bowls up to twelve of fourteen pieces can be accommodated, while in others only five to six pieces can be treated at once. What in many cases has not yet been realized, is that where the goods are treated in roll form, the number of pieces that can be treated at once is directly influenced by the pressure and tension placed upon the inner layers of the roll of pieces and that is the point at which faults begin to occur in the fabric. Thus is roll treatment of importance in the textile industry; for example, poplin cloths can rarely be crabbed in more than batches of four to six pieces at once, on the other hand some types of serges can be crabbed in batches of ten to fourteen pieces.

Roll treatment also demands equality of treatment, in that such a process as crabbing, both ends of the roll must be treated alike. This means that the goods must be reversed in their order upon the roller. This is generally accomplished by running the goods into the second bowl of the machine, the goods then being reversed in their order. This effectually assists in the effects at both ends of the roll being alike. One thing it does, but is often lost sight of, is that it emphasizes the folly of treating the goods in one bowl of the crabbing machine for by this method the effects at the piece ends can never be the same. The call for greater or quicker production has brought three and even multi-bowl crabbing machines. Yet it must be remembered that if goods are crabbed in three bowl machines the effects may be similar to only crabbing in one bowl of the machine. One thing stands out with regard to roll treatment of goods in crabbing, that if faults in the cloths are to be avoided, then the number of times that the roll of pieces is treated must be even if the roll of goods is treated more than once.

The same thing occurs during the steaming operations, for here the goods are wound under tension upon a perforated steaming can. Obviously the tension being constant, the pressure upon the inner layers of the roll is greater than that of the outer layers even if this is due only to the weight of the outer folds of cloth. It should also be remembered that due to increased resistance by the pressure upon the inner folds of the cloth, the steam has not as rapid penetration as when it reaches the outer folds of the roll. This often results in the portion of the fabric nearest to the steaming can, having more steam than the outer layers, thus often damaging the wool fibers of the fabric, through becoming brittle, etc., and thus the whole of the benefits of the steaming operation are lost. It should be remembered that there is a definite point at which steam may be passed through a fabric and that is when the wool fibers of the cloth cease to regain. If this point is passed then the fibers of the cloth are impaired and the benefits are lost. In addition, by no process can the wool fibers of the cloth be restored to their former state, once they are seriously injured during steaming.

Other points of importance in the steaming of fabrics in roll form are the temperature of the steam and the heat generated in the roll at the time of steaming. Obviously the tighter the goods are wound on to the steaming can, the greater the resistance to the passage of the steam, and also the increase of heat. There are instances where the fibers of the cloths have been ruined by this factor alone and they can never be restored to their original state. The practical man states that the goods must be steamed, either to avoid faults in the cloth, or to gain certain benefits in handle when the goods are finished. He is between two evils, if he gives the goods enough steam to ensure their permanent setting he is quite likely to impair the fibers of the cloth, or the temperature produced may be sufficient to yellow the goods and injure the fibers at the same time.

It should be stated that there is no need for goods during this operation to be wound upon the steaming can under extreme pressure or tension. In addition there is no need for violent steaming of the goods. The goods should in all cases where this is likely to occur be steamed lightly and then reversed in their order upon the steaming can and steamed a second time.

It has often been stated, that the operation of decatizing in finishing is very similar to that of the steaming process in the preparing of the goods for dyeing but it is very seldom that goods are twice treated or reversed in their order upon the decatizing machine in order to level up the

(Continued on page 466)

PROCEEDINGS OF THE
American Association of Textile Chemists and Colorists
AUGUST 12, 1935

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Members should notify the **Secretary** immediately of all address changes

Plan NOW to attend the Annual Meeting December 6 & 7 Chattanooga, Tenn.

COMMITTEES

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HEADQUARTERS — READ HOUSE

STANDARD SILK SAMPLES

There have been prepared, under the direction of the Sub-Committee on Fastness to Dyed Silk, standard dyed silk fabrics to represent four classes of fastness to washing—namely, Class I, Class II, Class III and Class IV. These standards have been carefully dyed with the dyestuffs and according to the dyeing methods recommended in the 1931 Year Book.

The A.A.T.C.C. is prepared to furnish sets of these washing standards for a nominal charge which will cover the cost of preparation. It will be possible to accurately grade the fastness by comparing it with the standards after subjection to the standard washing tests approved by this Association. All inquiries concerning these Silk Washing Standards may be addressed to the Chairman of the Research Committee—Louis A. Olney, Lowell Textile Institute, Lowell, Massachusetts.

Proceedings of the American Association of Textile Chemists and Colorists

Lubrication and Care of Textile Finishing Machinery*

By J. HILL ZAHN†

IN any talk dealing with a subject so broad as the lubrication and care of textile finishing machinery, we shall touch only the high spots, or those things which our organization feels so important that they are really essential.

First let me say that the care of textile finishing machinery is in many ways more important than the selection of the machines themselves. We may be able to do some satisfactory work with a poorly selected machine properly lubricated, but it is practically impossible to do acceptable work even with the best machines without proper lubrication.

Lubrication and Proper Lubrication

When I use the term "proper lubrication" I mean lubrication applied to bearings which have been properly prepared. Very frequently in improperly cleaning machines, dirt is washed down into the running parts, thus washing off the lubrication and impairing the efficient operation of the bearings. Many troubles encountered in finishing are due to improper lubrication; excessive horsepower is also due to the same cause. Recently we received a call from a mill having trouble in running a large calender—this calender was "looping back". In other words the cloth was hanging or bulging from the top roll. This part of the calender had been neglected and the bearings had apparently never been cleaned. After proper attention the calender was started and the trouble was entirely remedied.

A Series of Tests Showed This

Another case shows where excessive horsepower was used because of poor lubrication. We made a series of tests at one time with the old type of fixed lubrication. This at best is only partial lubrication. Then we carried on the test with automatic or continuous lubrication. The difference was amazing. There were nine 38-Cylinder Drying Machines in the test. After the installation of automatic oilers, the total amperes required for the electrical operation of the machines dropped from 320 to 188 and the approximate horsepower dropped from 47 to 28. The total cost at the time of the installation on nine machines was $684. Many times this cost will be saved in the reduction of electrical power required.

Lubrication means not only the application of the lubricant to the surface, but embodies cleanliness and watchful care of all driving or driven parts as well. Lack of proper lubrication means high coal or electric bills due to excessive consumption of horsepower and a consequently decreased profit—as well as often times seconds in cloth.

Cleanliness and Care

Of equal importance with lubrication is the necessity for cleanliness and care. For instance—starch boxes: these should be washed out regularly. When in use see that the starch is maintained at an even level and not allowed to overflow and waste. Dye Vats: here the importance of thoroughly cleaning and washing cannot be stressed too much. Just a trace of color carried over in the box when another color is being run will successfully ruin the shade.

Calender Rolls

Then take the all important subject of rolls. More things can go wrong because a roll has not been cared for than for almost any other reason. Before pointing out a few of these troubles let me run over the various kinds of rolls we have to deal with. They are rubber, wood, pressed rolls (cotton, combination husk, paper), brass, iron, steel, tube rolls of various kinds, etc. These demand individual treatment.

Rubber Rolls

Rubber rolls tend to wear down in grooves. They may have soft spots or "blisters." Other causes may make it necessary to turn them down. Here is where great care must be taken. There are several methods that might be used—depending upon the equipment selected. Some use ordinary machine lathes on which the necessary equipment is attached. In grinding, either an overhead drum type to drive the wheel from a wheel spindle or a tool-post grinder is used. If the tool-post method is used, it is recommended that the motor be sufficiently large in order to permit taking a sufficient cut into the rubber covering. We suggest at least a three horsepower motor, which would be ample for the work. This tool-post grinder is attached directly to the tool-post of the lathe. Rub-

*Presented at Spring Meeting, Piedmont Section, May 4, 1935.
†Manager, Southern Office, H. W. Butterworth & Sons Co.

ber covered rolls should always be ground dry, not wet. It is recommended that you use a wheel about 12 inches diameter by 1½ inches face. Wheels, the specifications of which are, Grit 40, Grade 3, Bond C-3-A are found to be most satisfactory. In grinding rubber covered rolls the traverse speed of the carriage or tool-post is approximately one inch per minute, or the slowest speed at which the lathe will run. The revolving speed of the roll being ground should be fairly slow or approximately 20 revolutions per minute. The wheel speed should be approximately 1800 to 2000 RPM.

This may appear to be rather slow work, but experience shows that in the long run it is best, because when faster speeds are used a burning effect is very apt to occur, causing the wheel to gum up and the surface of the roll to burn.

Grinding Rubber Rolls

Grinding rubber covered rolls in this manner will leave slight wheel marks on the surface. These marks can be removed in the following manner: Several plies of fine emery cloth should be mounted on a board, the object of the several plies being for a cushioning effect. This is held against the revolving roll, the speed of which is increased to the fastest speed at which the lathe will run, and oscillated across the surface by the operator, it is moved back and forth and not held stationary in any one place, which would cause a flat spot to result. This latter operation to remove the wheel marks can be efficiently done with a little practice by the operator.

Wood Rolls

When these rolls are not in use they will dry out and crack, and so become worthless, unless they are cared for. The usual method is to rig a perforated pipe over the rolls for their full length and allow water to trickle over them continuously. Some mills soak burlap or other materials and wrap up the rolls. Others have convenient ponds or troughs and keep them submerged.

Pressed Rolls

Here we have a slightly different condition to meet. These rolls, as most of you know, are built in hydraulic presses under pressures which vary from 500 to 1500 tons, depending on sizes, materials, use, etc. As these rolls run in the mangles and calenders the tendency of the materials out of which they are made is to work toward the ends. This causes the diameter of the roll to be slightly larger near the heads than it is in the middle. The consequence sould be obvious; the goods going through the machine will not be mangled (or calendered). The remedy is to turn down the ends of the rolls for a distance of 4 inches from the heads. The cut starts at 1-16" and tapers to 0".

Other troubles from pressed rolls occur where some foreign body is accidently passed through the nips. It is possible sometimes to avoid turning down the roll to eliminate the hole so caused, by putting the "dead set pin" in your levers at the top of the housings and "running in" the damaged roll. This is done by slightly wetting the roll with warm water, meanwhile running the calender. This treatment often brings up the material of the pressed roll and corrects the damage.

Brass, Steel and Iron Rolls

When anything goes wrong with these rolls about the only remedy is turning down in the lathe.

Tube Rolls

These are generally used as immersion rolls, carrier rolls, etc. It is absolutely essential that they be kept in perfect alignment. If they are "out" they will tell you about it themselves by putting the weft out of line. Then instead of looking for a weft straightener or other mechanical corrective, check up on the alignment of your rolls.

Running in Liquor

Rolls that run in liquor must be kept free running. Usually the bearings of these rolls lubricate themselves. Some bearings are located of necessity near starch boxes, dye vats, etc., and care must be used here to keep the lubricant from getting in the boxes or streaky dyeing will result.

I have mentioned only a few of the problems which regularly come up in connection with the operation of Bleaching, Dyeing and Finishing Machinery. Our organization will be glad to take up with you any individual condition which might arise in your own plant not only in connection with machines made by us but types other than we make as well. This is part of Butterworth service to the textile industry. We also have a booklet stating the kinds of grease and oils best suited to the lubrication of Butterworth machines. This was prepared by one of the oil companies after a complete study of Butterworth machines and the work they are designed to do. If you would like to have this booklet I shall be glad to see that it is sent to you.

DISCUSSION

R. W. Arrington: I want to ask about the speed of tenters. Tell us something about these so called "fancy" tenters and high speed tenters.

Mr. Zahn: This is rather a complicated question. I often wonder when the speed of tentering machines is brought up if any consideration is given to the class of fabric the machine is to handle. I know of a number of tenters that are operating satisfactorily at a speed of 120 to 140 yards per minute, but would say that the average speed of tenters is approximately 100 yards per minute. As far as I know, 150 yards per minute is the fastest running tenter in this section. However, we can furnish a tentering machine that mechanically will run 200 yards

per minute. The only difficulty we have encountered in this high speed running has been in the finish of the fabric handled.

Mr. Arrington: Tell us something about speeding up various machines. Can this be accomplished on the old type machines?

Mr. Zahn: The tendency today is to speed up all machines. On the latest designed machines this has been taken care of by improvement in construction and the use of anti-friction bearings. In many instances where certain machines were running 30 to 40 yards per minute they have been "stepped up" to approximately 200 yards per minute. Some were old machines rebuilt while others were replaced with entirely new machines. I think sometimes this question of speed is stressed too much, and I would call your attention to the fact that some plants have found it necessary to reduce speed because they ran into trouble securing the desired finish on their fabrics.

Mr. Arrington: Let me ask you another question—it may not be in order. I think with all of us machinery goes in "style"—like clothes, etc., and there is a lot of advertising today about individual drives being far superior to range driven finishing machinery. What does the trend of time show in contrasting the range finish to individual machine finish?

Mr. Zahn: At the present time there seems to be a greater demand to finish with units; while a few years ago plants were installing equipment for handling greater yardage and very often a range was furnished consisting of anywhere from five to fourteen machines. Today very few plants are interested in these long ranges. This I think is due primarily to the change in the quantity of work they are now handling. I do not say that individual machines are not in demand; however, most of our inquiries are for "hooking-up" two or three machines.

Mr. Arrington: In some plants has not the trend gone too far?

Mr. Zahn: Yes. I think some finishing plants have synchronized too many machines, and at the present time are changing these over to unit ranges. Long ranges were installed when they had a large run of one class of goods. Today's market evidently is not calling for large runs—in the average plant I mean. However, a few plants still specialize in one line of work and of course the only way that they can economically operate is through the use of long range machinery.

H. D. Learnard: I cannot ask a question, but I can confirm what has been said. A great deal depends on the care of machinery. I was interested in the statement about washing machinery; grit washed into bearings will a few days later cause trouble.

In reply to one of Mr. Arrington's questions about the speed of tenters, I would say that we have been able to run along at about 143 yards per minute, but not beyond that—the drying of the goods enters into the problem. The machinery itself—the tenter and chain—would stand considerable more speed, but if you drive it faster there is difficulty with brittleness in too rapid drying.

We have calenders geared to run 300 yards per minute but we keep them down to approximately 260 yards per minute. There is difficulty with the operators—when the tone of the machine changes they seem to be afraid of it, and some one in authority has to stand by and see that they run at that speed. If left alone at any time we find they slow down. There is no difficulty in running from 200 to 210. Up to that it is all right, and of course habit has something to do with it. They've been accustomed to a certain tone and any other tone alarms them as they think things are going wrong.

Question: Along the line of roller bearings—is the horse power reduced when you put in roller bearings?

Mr. Zahn: Yes, considerably reduced in the same speed and same style of cloth. The speed with plain bearings is about 180 yards per minute and the horsepower 65. By putting anti-friction bearings in, the horsepower is reduced from 65 to 36½.

C. L. Eddy: I should like to ask Captain Learnard a question. Do you know whether your damages were increased when you jumped from 120 to 220 yards a minute?

Captain Learnard: Yes, at first they were—due to the rapid stretching of cloth, but we took care of this by putting the cloth through our tenter frames and pulling it back to width. It has been our practice to go through starch range and then to drying cylinders . . . through tenter to finish drying and bring to width. We have skipped the drying cylinders to go right into the tenter frame.

Mr. Schroeder: I have trouble getting drain valves and valves used on dyeing machines to stand up.

Mr. Zahn: I am sorry, but I can't be of any help to you. Your trouble is evidently due to chemicals eating valves away.

Tom Smith: There is a valve on the market that will withstand chemicals. It is all cast iron and is a quick opening and closing gate valve.

CALENDAR OF COMING EVENTS

Annual Meeting

December 6th and 7th

Read House, Chattanooga, Tenn.

Proceedings of the American Association of Textile Chemists and Colorists

The Chemical Industry*

By H. L. DERBY†

I HAVE very much enjoyed participating in the festivities of this occasion and feel particularly honored in the invitation, extended through Mr. Haddock, to speak to the Piedmont Section of the American Association of Textile Chemists and Colorists, on the occasion of its Annual Outing.

The chemical industry, of which you are a part, has contributed in no small degree to the development of this section and other sections of the South in which industry has made tremendous progress in the last two decades.

Without the contribution of the chemist, and in that I include of course the color expert, the great textile mills of our country would find no market for their products.

The chemical industry of America, prior to the World War, was not developed in this country as in foreign countries and the nation was obliged to import not only the major part of its colors but many of its essential chemicals. The demands of war brought about a changed condition, and with peace came a full realization of the necessities if America was to be self-contained in this important branch of industry.

During the last 5½ years we have gone through a period of intense depression. A series of economic events having tremendous force and gaining momentum, which it was impossible to control, brought about the necessity for readjustments not only in our economic life but in our social activities as well. That our country has survived and our institutions have been preserved is a tribute to the character of our citizens. We find ourselves in a state of uncertainty and with many conflicting opinions and ideas as to the course which in the immediate future should be pursued.

Our industry, I feel, has given a good account of itself. During the last few years some industries have operated at 15 to 20 per cent, and some even less, with thousands of their former employees on the public dole. The chemical industry has gone through this period with an average of employment well above 75%. Through research and development, new products of chemistry have been perfected and these products, being adapted to commercial use, have provided employment for thousands of men. As usual in a period of depression, the demand arose for low-priced merchandise—quality being one of the lesser considerations,—the reverse to the prosperity type of demands. In past generations the distinction between good and cheap merchandise was readily discernible by the most inexperienced observer; however, modern chemistry has largely eliminated the superficial differences by neatly and attractively covering up the inferior quality.

The chemical industry is basic and there is scarcely a product manufactured in any industry to which the chemist does not make a contribution. As necessity has been the mother of invention, it will be the mother of chemical application.

A striking illustration of the chemists' contribution to defense and the safeguarding of lives in the World War was the production of a gas defense mixture. When the Allies were first subjected to a gas attack, they frantically strove to produce a material which would combat this death-dealing agent. For nearly two years prior to America's entry into the conflict, a defense mixture was furnished by America for the Allies' gas masks and when the United States finally did send her soldiers to the front they were completely protected by their masks, the records showing that not one fatality resulted from a gas attack to those soldiers who wore their masks. It would take hours to tell of the contributions of the chemist to the welfare and security of Society.

In 1914 the chemical industry of America employed 249,000 persons,—today there are in excess of one-half million men employed in our chemical industries. The contribution of the industry to society's income, in wages and salaries, today is in excess of $700,000,000, annually. Prior to the war the total capital invested in the chemical industry, was less than five hundred million dollars and it is now in excess of two billion dollars. This growth was financed conservatively, as is evidenced by the fact that the bonded indebtedness of the chemical industry bears a lower ratio to total assets, plant investment and gross income than any other leading manufacturing industry.

The chemical industry has a large financial stake in Government. It contributes in excess of $200,000,000 per annum in Federal, State and local taxes, and this is equivalent to 25% of the total taxes paid by all other manufacturing industries combined. This tremendous tax bill

*Address at Annual Outing, Piedmont Section, June 29, 1935.
†President, American Cyanamid & Chemical Corp.

results to some extent from the fact that the industry has obtained a better demand for its products and their earnings have accordingly been better sustained. Corporations making no money are not required to pay income taxes, but the taxes on such industries as the chemical industry, where earnings have continued, have been levied as a disproportionate share of the tax burden. This fact alone is sufficient warrant for those of our industry to take a leading interest in the demand for reduced Governmental expenditures and in the operation of Government itself.

There is no other industry in America that has spent so much money during the last five years on replacements, repairs and extensions as has the chemical industry—the aggregate being well over $50,000,000 in 1934 alone. That there have been no major labor disturbances in the chemical industry speaks well for its management. It has spread employment in its plants; kept ahead of or apace with any general increase in labor rates and has done its part to relieve conditions of unemployment and depression. Had it been possible for all other industries to do as well in the matter of employment, as did the chemical industry, we would have no great unemployment problems today.

No country has ever escaped some penalty for extravagance or for a program of spending beyond its resources and income. Government debt is increasing at the rate of $6,500 per minute, and that tremendous sum must be paid by the taxpayers of this generation or some future generation. The latest estimates of the tax burden in the United States, on the basis of population are:—

1933-34 ..... $75.14 per Capita
1934-35 (about) ..... 79.00 per Capita

We are more heavily taxed per capita than the French and Germans, and with the taxes in the United Kingdom decreasing from $91.81 in 1933-34 to $87.50 in 1934-35, we will soon be more heavily taxed than the British.

We, as a nation, are engaged in a series of gigantic experiments; some of these experiments have been tried in other nations and abandoned. Others have been persisted in with results which are well understood. There are many of these experiments which are in the process of being rejected and others which may suffer the same fate.

The effect of a processing tax is well known to you all.

The Supreme Court has recently declared the N. I. R. A. to be unconstitutional. Other changes will no doubt take place.

There is no other nation on earth possessing a market so desirable as that of the United States. With its 126 million people, with an average wage level in excess of any other country on the globe, and with the highest standards of living in the world, we are in position (as is no other nation on earth) to be a self-contained nation, should we so elect. Yet, notwithstanding this tremendous advantage, we find some who would trade this market for the poverty-stricken markets of the world.

I hold no brief for excessively high tariffs but I do not think we should overlook the facts of the tariff history of America. The First Continental Congress enacted tariff legislation and laid down the principle that the growth of the nation was dependent upon encouraging and promoting agriculture and the manufacture of goods in this country. Our forefathers did not go to the extent of subsidizing producers, but set up a limited tariff protection against foreign importations on those articles (few in number, at that time) which it seemed possible to produce and manufacture with home labor.

From time to time readjustments in tariff rates and changes in administrative provisions had their culmination in the present Law, enacted in 1930, and known as the "Hawley-Smoot Act," or "Public Law No. 361." That law has for its purpose "to provide revenue, to regulate commerce with foreign countries, to encourage the industries of the United States, and to protect American labor, and for other purposes." Congress endeavored to fix schedules which, as far as could be foreseen, would equalize the difference in cost of production at home and abroad. There was provided in the Act by the so-called "Flexible Section" which makes it possible for the President to raise or lower any duty within the limits of 50%, provided the U. S. Tariff Commission finds such change is necessary to correct any inequality which may exist. Although there are some 6,000 articles covered by our tariff, less than 200 rates of duty have been attacked as being too high, and less than 50 cases have been decided in favor of the proponents, which, it would seem, speaks well for the painstaking care of the Congress in preparing this law.

This act has now been amended (in 1935) to provide, among other things, that the President may arbitrarily lower rates to the extent of 50% to encourage reciprocal treaties with foreign nations. The legislation was described as being of the "emergency" kind, but not withstanding that fact, the amendment to the tariff law provides that the proposed Foreign Trade Agreements "Shall be subject to termination upon due notice to the Foreign Government concerned, at the end of not more than three years from the date on which the Agreement comes into force, and if not then terminated shall be subject to termination thereafter upon not more than six months' notice." It will therefore be seen that the amended act provides that these tariff bargains may extend for a period of 3½ years, or longer, even though the "emergency" may have long since ceased to exist.

The opponents of this amendment objected, for two principal reasons: First, that the executive branch of the government—independent of Congressional action would be empowered to change the rates of duty fixed by Congress, without Congressional review, and, Second: That

a duty might be cut in half even though that duty was not higher than necessary to equalize the cost of production abroad and at home. In other words, in these so-called "Trade Agreements," under the Reciprocal Plan, a foreign country may make a trade to permit shipping foreign goods into this market and enjoy a cost advantage so great (with tariff protection reduced by one-half) as to destroy the business of the American manufacturer. There is no appeal from these treaty agreements.

The opponents of the measure protested that tariff-making had ever been a function of Congress and that duly elected representatives of the people should have the responsibility and the function of fixing tariff duties. Those in favor of the provision argued that Congress was not surrendering its prerogatives in tariff-making but was simply delegating to the President and the executive arm of the government the power to carry out its will, and that its will included the Foreign Treaty Trade Agreements provided for in the amendment to the act.

So far, the treaty agreements which have been concluded by the Department of State are those with Cuba, Brazil, Belgium, Haiti and Sweden, but there are a large number of other treaty agreements under negotiation and these will no doubt be consummated in the near future. At this time it is impossible to summarize the advantages or disadvantages of these agreements. There have been vigorous criticisms levelled at those already made, and the opposition prophesies heavy losses and many disadvantages to American producers. The lack of any advance knowledge by those who may suffer as to what the agreements may embody, and the futility of protest when the agreements are concluded, creates a situation heretofore never experienced in American tariff-making.

The law also provides that "the proclaimed duties and other import restrictions shall apply to articles the growth, produce or manufacture of *all* foreign countries, whether imported directly or indirectly." Thus, may an agreement be made with one foreign nation, but *all* foreign nations having commercial treaties with the United States may benefit by it without any reciprocal advantage (anticipated or real) accruing to producers in the United States.

It seems to me there are two things fundamentally wrong with this process of reasoning: First, the importation of any foreign goods which can be manufactured in this country deprives American labor of the work required to manufacture these articles. Second: Our favored nations treaties make it obligatory that wherever a tariff is reduced in favor of one nation, that reduction on that article applies to every other nation with whom we have Trade Treaties, whether or not a reciprocal agreement is being negotiated or will in the future be made. For instance, if it were possible to make a treaty with France whereby they purchased wheat and lard in exchange for being allowed to ship us textiles, our treaty with Japan permits her to ship her textiles to this country at whatever duty is arranged with France, and without any obligation on the part of Japan to take any wheat or lard.

The outcome of this gigantic experiment is not looked upon with confidence by those fully realizing its possible results. Only time will tell the extent of profit or loss resulting from this radical departure in our tariff-making. That this plan does not encourage investment of capital in industrial enterprise, is putting it mildly.

It is hard for those who are adversely affected to understand what advantage accrues to American labor and industry when one home industry is closed down or seriously curtailed, to permit a balance of trade in favor of a foreign country, in the hope that that foreign country will use that balance of trade to purchase the products of some other industry in this country.

About seventy percent (70%) of the imports of the United States come in duty free, and tariff rates are only assessed on those articles which are produced in adequate amount and quality in this country. In other words, our theory has been to purchase those goods needed in this country and impose no tariff or restrictions on their importations, but to tax those foreign goods which are not needed in this country, at least to an extent necessary to equalize the domestic production cost.

It is argued that the foreign countries will not buy our merchandise unless it be better and cheaper than they can purchase elsewhere, and that no reciprocal treaty will result in their purchasing an excessive quantity of the goods we manufacture or produce. It is easy to understand why the foreign nations should be willing to trade their more or less poverty stricken markets for the unequalled American market.

A plan to throw men out of employment in one industry in the hope of benefiting another industry, is an extreme innovation. It was held by some proponents of the bill, when Congress was giving it consideration, that the intention was only to curtail "inefficient" American Industries. This would, they said, permit foreign countries to enjoy our markets for goods which were not "efficiently" manufactured here and in turn this increased outlet for foreign merchandise would result in the "efficient" American producer increasing his sales abroad. To whom should the power be delegated to judge the degree of efficiency of an industry? In the manufacture of many new products, particularly in the chemical industry, it is impossible to become efficient, except with experience, and with money spent in research and development. If "efficiency" alone is to be the measure of the life of our industries, then the progress which we have made in the past must cease and new developments, new products and general advancement in manufacturing science must cease.

Recently, the Department of Commerce issued a bulle-

tin on world chemical developments in 1934 and one striking fact brought out in that report shows that the average laborer in the chemical plants in Japan receives 57.9c per day, for 11.31 hours. The report further shows that the average rate of wages in chemical plants in Germany is 20% less than in 1930 and, at the present rate of exchange, these workers are paid between 20 and 25c per hour. The present average wage rate in chemical plants in the United States is in excess of 50c per hour.

No argument is necessary to convince any fair-minded person that a cost equalizing tariff has not and will not create a monopoly in this country. It seems equally well established by an examination of our history that the tariff plays no small part in our high scale of wages and standards of living. Should our Government persist in this new and untried experiment, with its radical departure from successful experience, and invite consequences which may entail irreparable losses to our national welfare?

I believe in the future of America. I believe in the sanitary and wisdom of its people. I believe that organizations such as this have a responsibility and duty to those who have not the same opportunity or the same ability to think and reason through the situations which confront our Nation.

With all the pride you have in the Institutions of America, with all the patriotism that is inherent in you, it is your duty and mine as Americans to steadfastly stand for the things which are American and defend America from all misguided, though well meaning theorists, who would substitute experiments which, at best, will advantageously affect only a small minority of our citizens, for the lifetime experience of progress and improvement that this nation has enjoyed through its history.

ANALYTICAL METHODS
for a
TEXTILE LABORATORY

Our supply of copies of this publication by Walter M. Scott has been exhausted.

H. C. CHAPIN, Secretary

UNEMPLOYMENT REGISTER

The following application is in addition to those which were published in the issue of July 1, 1935. See that issue for details of this department.

A-B-E-F-I

Education:—Ph.D. in Chemistry, Pennyslvania, 1927; Graduate work in Chemical Engineering, M. I. T., 1918.

Experience:—Five years teaching, eleven years industrial and consulting experience on cellulose, synthetic fibers, dyes, textiles, oils and soaps. Now head of Chemistry and Dyeing Division of leading textile school. Age 45 years, married, member Alpha Chi Sigma and Tau Beta Pi, publications, references. Now on summer vacation.

ANNUAL OUTING, PHILADELPHIA SECTION

The Annual Outing of the Philadelphia Section was held at the Torresdale Country Club on Friday, June 7th. There were 230 members and guests present. There were golf activities for those so inclined and other games for those not golfing. This was followed in the evening with dinner and entertainment.

The following officers were elected for the coming season: Chairman, Robert E. Sullivan; Treasurer, William Ebersole; Secretary, R. W. Delaney.

GROUP PHOTOGRAPH, PHILADELPHIA SECTION, TORRESDALE COUNTRY CLUB, June 7, 1935

AMERICAN
DYESTUFF
REPORTER

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Published biweekly by
HOWES PUBLISHING CO., INC.
440 Fourth Avenue, New York City

Subscription price: $5.00 per year; Canadian $6.80; foreign $6.00

Vol. XXIV August 12, 1935 No. 16

ON GETTING ALONG WITH PEOPLE—IV

(Continued from last issue)

Running a Mill Experiment

RUNNING a Mill Experiment may seem a little broad as a subject but there are general principles that are common to about all kinds of mill experiments, especially when they involve working with other people.

We will start with a somewhat unusual preliminary classification. There are two sorts of experiments: (1) those that you have proposed yourself, and (2) those that have been suggested or ordered by somebody else.

Until you are a well recognized part of the organization the first kind may require much care and thought in order that they may be carried out properly and not get you into trouble. The responsibility is usually greater if you are carrying out your own ideas than if you are trying out those of other people.

The experiment may be a simple routine matter hardly deserving that name, but mill experiments are likely to require very precise conditions and must take many factors into consideration if they are to be entirely successful.

One of the commonest cases is that of getting samples put through a mill and whether bleaching, dyeing, printing or whatnot, it offers technical and human problems which combine to make it a very interesting subject for study. For the present purpose we assume that the technical matters are in the capable hands of the laboratory and of the mill executives but, as is likely to be the case, that the human relationships are not so well in hand.

Perhaps also we should explain that we are not thinking so much of the routine sampling which may be a regular and necessary part of a mill's work as of such miscellaneous items as represent attempts at process economy or improvement, tests on new materials and matters that are of a research nature.

It usually happens that such samples whether a single skein or a regular lot of yarn, a swatch of cloth or a full sized piece of roll, usually interrupt somebody who is doing something else. More frequently they interrupt or delay more than one person, the foreman and one or more operatives. Naturally, then, certain amounts of care and thought are necessary in approaching such a matter if good results are required. It may be a recognized duty of the mill department to put through such tests, but each specific request partakes very much of asking a favor.

In the first place the result is usually wanted *now* rather than at the department's convenience. Cleaning out a machine, or new settings may be necessary and it is a good foreman or operative who is willing to have his production schedule interrupted or his record impaired by odd jobs which may mean nothing at all to him personally.

Now foremen, just like any other group of people selected from the population at large are of all kinds. They vary from the high and mighty know-it-all to the "regular fellow" who not only takes a general interest in seeing an experiment go right, but may even be able to learn more from it than its sponsor.

It is at least an uncommon foreman or superintendent who does not like to know what you are up to if you are doing something in his department. It is certainly the best ethics to take your problem directly to the man in charge, and even if he says to just go ahead and have your material put through it is wise, and satisfying to him, if you drop around with a word as to what is under way.

From the experimenter's point of view it is often much better to insist, if possible, that the foreman "stick around." He can frequently point out things that might not otherwise be noticed, like some defect in the conditions of the experiment which would make it impractical, or some flaw in the resulting goods outside the immediate purpose of the experiment.

And, another thing, if the result is not clear or not complete at the time of the experiment step around later if the matter is not extremely confidential, and tell the superintendent, foreman or even the machine operator how the experiment came out. I know from much experience that the good men on the job are interested, if they understand their work at all, and they can easily be made to feel that "we" did it which is usually the truth, and to have a real interest in the next experiment, which is a great advantage.

Don't abandon an experiment part way through without giving an explanation. If you do that very many times, (and it is often clear soon after starting that an experiment may not be of any use) you will build up the impression that your experiments do not mean much and are not important. If they actually are not important don't trouble other people to make them; if they are important look after them and explain any discontinuance.

To inspire interest and confidence in your work it is more than likely to be advisable to go over your project or experiment carefully beforehand with the foreman, subforeman, or perhaps the workman who is to run it. You not only want to be sure that he understands what is

wanted but you need his interest, and he is more likely to take necessary precautions and watch details if he is taken into your confidence as far as permissible.

Next time we will be more specific about some of the troubles that beset mill experiments. While any example that may be given may sound rather simple, actually there are few pursuits that require more alertness than running mill experiments.

(More comment on this subject will appear in our next issue.)

New Patents

(Abstracted by S. O. C. M. A.)

Dyestuffs of the Dibenzanthrone Series. (Dye vegetable fiber usually violet, dark blue to olive and grey shades; for example, comprises reacting dibromodibenzanthrone (prepared by brominating dibenzanthrone in chlorosulphonic acid in the presence of sulfur) in a mixture of nitrobenzene, copper carbonate, sodium carbonate and 1-aminoanthraquinone; a continuation in part of copending application Ser. No. 246,078, filed Jan. 11, 1928.) Max Albert Kunz, Mannheim, and Karl Koeberle, Ludwigshafen-on-the-Rhine, Ger., assignors to General Aniline Works, Inc., N. Y., N. Y., No. 1,994,136, March 12, 1935.

Azo-Dyestuffs, and Process of Making Same. (For example, comprises coupling the acid sodium salt of 4-nitro-4'-amino-stilbene-2.2'-disulfonic acid with salicylic acid and then treating with caustic and glucose; dye cotton bright orange shades fast to light and chlorine.) Hans Schindhelm and Richard Gast, Frankfort-on-the-Main-Fechenheim, Ger., assignors to General Aniline Wks., Inc., N. Y., N. Y., No. 2,004,250, June 11, 1935.

Azo-Dyestuff and Fiber Dyed Therewith. (Obtainable by combining, in substance, on the fiber or on a substratum, the diazo-compound of 5-chloro-1-aminobenzene-2-sulfonic acid dimethylamide with 2,3-hydroxy-naphthoic acid anilide; when produced on the fiber yields a golden-orange shade of a very good fastness to light.) Kurt Schimmelschmidt and Hans Thomae, Frankfort-on-the-Main, Ger., assignors to General Aniline Works, Inc., N. Y., N. Y., No. 2,004,383, June 11, 1935.

Vat and Sulfur Dyestuff Preparation for Printing Cotton. (More specifically, comprises 6.6'-diethoxy-2.2'-bisthionaphtheneidigo and a compound selected from the group consisting of anthraquinone-1 and 2-mercaptan and the corresponding disulfides.) Hermann Berthold, Leverkusen-I. G. Werk, Ger., assignor to General Aniline Wks., Inc., N. Y., N. Y., No. 2,004,458, June 11, 1935.

Sulfonic Acid. (Dyestuff intermediates obtained by subjecting diphenylbenzenes to sulfonation by means of chlorosulfonic acid, sulfuric acid or acetosulfuric acid.) Carl Wulff and ESrnst Roell, Iudwigshafen-on-the-Rhine, Ger., assignors to I. G. Farbenindustrie Aktiengesellschaft, Frankfort-on-the-Main, Ger., No. 2,004,546, June 11, 1935.

N (p.Amino-Phenyl) Morpholine and Method of Preparing the Same. (A new chemical compound for use as a dyestuff intermediate, obtained by action of zinc dust on hydrochloric acid solution of the p-nitroso compound.) Herbert A. Lubs, Wilmington, Del., assignor to E. I. duPont deNemours & Co., Wilmington, Del., No. 2,003,763, June 11, 1935.

Dyestuffs of the Anthraquinoneacridone Series. (Namely, 4-amino - Bz2,3,5-trihalogenanthraquinone - 2,1 (N)benzacridones and their acyl derivatives; vat dyestuffs equally dyeing viscose silk and cotton, violet to blue to green blue and green gray shades.) Max Albert Kunz, Mannheim, and Erich Berthold and Karl Koeberle, Ludwigshafen-on-the-Rhine, Ger., assignors to General Aniline Wks., Inc., N. Y., N. Y., No. 2,005,321, June 18, 1935.

Dyeing Composition. (Comprises a mixture prepared from diazo-amino compound composed of tetrazotised dianisidine and amino-sulfo pyridine and Naphthol AS, yielding a blue shade when applied on textile.) Eugene A. Markush, Jersey City, N. J., assignor to Pharma Chemical Corp., N. Y., N. Y., No. 2,005,347, June 18, 1935.

Dyestuffs of the Dibenzanthrone Series. (A division of above patent No. 2,005,529.) Alexander J. Wuertz, Carrollville, Wis., and Oakley M. Bishop, deceased, late of Wilmington, Del., By Eva P. Bishop, Wilmington, Del., and Wilmington Trust Co., Wilmington, Del., executors, assignors to E. I. duPont deNemours & Co., Wilmington, Del., No. 2,005,574, June 18, 1935.

Dyestuffs of the Dibenzanthrone Series and Process of Preparing the Same. (From certain mono-alkyl ethers of dihydroxydibenzanthrone; novel vat dyes of the Jade Green series, which can be used directly as vat dyestuffs converted by further treatment into acid wool or silk dyestuffs, or again into dyestuffs suitable for dyeing cellulose acetate fibers.) Otto Stallmann, So. Milwaukee, Wis., assignor to E. I. duPont deNemours & Co., Wilmington, Del., No. 2,005,810, June 25, 1935.

Water Insoluble Azo Dyestuff Containing a Benzanthrone Nucleus. (Namely, naphthols, 2-hydroxynaphthoic acid arylamides, hydroxy-anthracene-o-carboxylic acid arylamides, etc., obtainable by diazotizing Bz-1-amino-benzanthrone or a 2-alkoxy-substitution product and coupling in substance, on a substratum or on the fiber with a coupling component, yielding, when produced on the fiber, various shades of good fastness to boiling and chlorine.) Gerhard Schrader, Opladen, near Cologne-on-the-Rhine, Ger., assignor to General Aniline Wks., Inc., N. Y., N. Y., No. 2,005,848, June 25, 1935.

4-Amino-1.8-Naphthalene Dicarboxylic Acid Imides and 4-Alkylamino-1.8-Naphthalene-Dicarboxylic Acid Alkylimides and a Process of Preparing Them. (Dyestuffs and dyestuff intermediates, obtainable with a very good yield and in a very pure state by heating 4-sulfo-1.8-naphthalene-dicarboxylic acid with cyclohexylamine, alkylamine, hydrazine, ammonia or an agent yielding ammonia.) Wilhelm Eckert and Walter Gmelin, Frankfort-on-the-Main-Hochst, Ger., assignors to General Aniline Wks., Inc., N. Y., N. Y., No. 2,006,017, June 25, 1935.

A New Method for Distinguishing Between New and Second-Hand

Cotton Filling Materials

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INTRODUCTION

THE Division of Bedding of the New York State Department of Labor is concerned with the enforcement of Article 25-A, General Business Law. This Law provides for proper designation of contents and type of fillings of bedding and upholstered furniture sold in the State of New York.

One of the principal functions of the laboratory of the Bedding Division is to distinguish between new, old and second-hand materials used in filling articles of bedding and upholstered furniture. It is the practice of some fraudulent manufacturers to use second-hand materials and designate the contents as new. This practice is illegal and a proper analysis of the contents is essential to insure the purchaser that he receives merchandise he intended buying.

In connection with the studies herein embodied, the term "cotton filling" is used to indicate the resilient fibrous material used inside an article of bedding or an article of upholstered furniture. It may be in the form of blown and compressed cotton fiber aggregates or as a felt made of layers of garnetted cotton fibers.

Prior to the establishment of technical methods for the distinction between new and previously used cotton fillings, the method generally used was visual examination of the material. This examination was usually based on intuitive rather than exact methods. The first scientific method was suggested by Winne & Donovan[1] of the Maryland Health Department in 1932. They found that cotton fibers emitted characteristic fluorescences when exposed to rays of ultraviolet light of definite wave length. New cotton fibers give off a purple or blue fluorescence, while second-hand fibers produce ivory white or tan fluorescence. Shortly after this, Burke and Kane[2] of the New York State Dept. of Labor developed a chemical test to differentiate between new and second-hand cotton fillings. They found that new cotton fillings had a water-soluble sulfate content of less than 0.25% expressed as Na_2SO_4 and that second-hand cotton fillings were characterized by a sulfate content of more than 0.25%.

When the laboratory of the Division of Bedding was established in 1933, these methods were further investigated, and the results obtained warranted the acceptance of the two tests as the standard scientific methods for the differentiation of new from second-hand cotton fillings.

These tests served satisfactorily until a short time ago when economic conditions forced suppliers of cotton waste fibers to go to foreign markets for them. Analyses of samples of representative new foreign wastes showed that many of them had sulfate contents above 0.25%. It was later learned that some foreign wastes before shipment to this country are fumigated with sulfur or sulfur dioxide gas, or are loaded with clay or hygroscopic alkaline earch salts, chiefly chlorides and sulfates,[3] the latter to prevent loss of moisture, keep the fibers pliable, and increase the weight. This introduction of sulfur, sulfur dioxide, or soluble sulfates has a pronounced effect upon the soluble sulfate content which, under these conditions, can result in erroneous conclusions as to the newness of some of these materials. It was also found that some of these wastes had a fluorescence resembling that of previously used material. It was subsequently determined in this laboratory, that conditions of exposure affect the fluorescence of cotton fibers. A sample of cotton linters exposed for six weeks to air saturated with water vapor changed in fluorescence from the purple of new material to an ivoryish tan with a few purple and blue spots commonly associated with doubtful and probably previously used material. The suspicious fluorescence of some of the foreign wastes examined may therefore, be attributed to the climatic conditions encountered in India and China and also during transport to the United States.

Due to the foregoing disturbing influences on the differentiation of new from second-hand materials, the authors undertook to determine a characteristic of second-hand cotton fillings which would not be affected by any of the described treatments to which the material may have been subjected.

As previously indicated, Burke and Kane found that second-hand material showed an increase in sulfate content. It was further determined by one of us (W. L.) that new cotton fibers do not contain enough sulfur in any form to account for this increase. It was concluded that the additional sulfate found in second-hand cotton fillings must have come from the outside during the period of its use, probably from perspiration or urine. It is likely that bedding and upholstered furniture fillings would become contaminated with the constituents of ani-

*Bedding Division Laboratory, New York State Department of Labor. Publication approved by L. A. Havens, Director, Bedding Division.

[1] Winne, A. W. and Donovan, J. D.: Amer. Dyestuff Rep., *21*, 601 (1932).

[2] Burke, W. J. and Kane, H. J.: Unpublished data.

[3] Barnes, J. H.: J. Soc. Chem. Ind., *35* 1191 (1916).

mal waste fluids on prolonged use. The normal adult in twenty-four hours excretes over 30 grams of urea in urine[4] and perspiration[5] and also several grams of ammonia and other nitrogenous compounds in these fluids. It was decided to attempt to differentiate between new and used cotton fillings on the bases of their content of these end products of nitrogen metabolism.

To carry out the analysis for urea, a modification of the urease method developed by Van Slyke and Cullen[6] for the estimation of urea in blood and urine was used. Ammonia was determined by the usual micro-Kjeldahl method. After determining the proper conditions for these analyses, representative samples of materials used for the manufacture of new cotton felts and of known second-hand cotton filling materials were analyzed. The results are given in tabular form elsewhere in this paper.

PROCEDURE

One cc. of 0.1N HCl is added to the aqueous extract from 5.00 grams of the cotton to be tested and then concentrated to a volume of 4 to 6 cc. This is washed into a graduated tube, one cc. of 0.1N NaOH added, and the volume made up, after cooling, to 12.5 cc. 5 cc. aliquots are pipetted into each of two large test tubes.

The first (I) is buffered to a pH of about 7 for maximum hydrolytic activity[7] and a urease tablet is added. This is allowed to stand for 15 to 20 minutes at room temperature. The contents of the tube are made alkaline with borax solution, the test tube is then introduced into the distilling apparatus* and the liberated ammonia caught in 2% boris acid containing a few drops of methyl red indicator solution. The liberated ammonia is titrated with 0.01 N H_2SO_4 to a deep pink; the flask is heated to boiling, and the titration continued hot until a full red color is reached.

The second aliquot (II) is made alkaline with borax solution and the liberated ammonia distilled off and titrated as described above. A pair of blank determinations must be run with distilled water and the reagents alone. The titration values from the blanks are subtracted from the values obtained in the analysis of a sample to get the corrected volumes to be used in calculating the results.

Calculations:

% NH_3=corrected vol. in cc (II) x 0.0085

% Urea=corrected vol. in cc (I-II) x 0.015

DISCUSSION

Examination of the data in Table I shows that the highest percentages of ammonia and urea in new material are 0.023 and 0.009 respectively. The data in Table II shows that the percentages of ammonia or urea in second-hand materials are generally above these figures. The values of 0.03% for ammonia and 0.01% for urea were arbitrarily chosen as the dividing lines to differentiate between new and second-hand material. All the new samples fall well below these values while of the second-hand samples analyzed 85% had ammonias above 0.03% or ureas above 0.01% or both. The remaining 15% of the previously used samples had ammonias and ureas of the same magnitude as the new samples. This may be attributed to use of the filling for a short period, or use in such a manner that it is well protected from contamination, and while technically second-hand, would show little effect of use. While by this test the distinction between new and second-hand cotton fillings is not absolute, no new material will be called second-hand by this method, even though previous tests might have done so. Since the mislabeling of cotton fillings is a penal offense in the State of New York, it is far preferable to indicate a few second-hand specimens as new than to designate even a single sample of new material as second-hand and so prosecute an innocent party.

This laboratory in determining if a sample of cotton has been previously used, uses the results of these test along with the sulfate determination and the ultraviolet fluorescence. In the hundreds of cotton specimens examined subsequent to the data cited in this paper there was never found a sample with a high ammonia content along with a blue fluorescence and a low sulfate. In every case of a high ammonia or urea content there was also found a high sulfate and an ivory or tan fluorescence. This eliminates the possibility of finding a sample with a high ammonia content due to contamination from sources other than animal bodies. The many samples of new cotton which have been examined were from all the cotton waste sources, foreign and domestic, shipped and stored under various conditions. It is highly improbable that *only* such specimens which already had *both* a high sulfate and an ivory or tan fluorescence would become contaminated with ammonia from non-animal sources

To determine whether the processing of cotton wastes into resilient filling materials affects the urea and ammonia tests, two samples of cotton felt of known composition together with their components were obtained. No. 1 was a felt made up of 2-3 India picker and 1-3 India fly. No. 2 was a felt made up of 50% India picker, 25% domestic fly and 25% China fly. The felts and components were subjected to the analyses described and results tabulated in Table III.

The results of the analyses in Table III show that the ammonia and urea in the felts are approximately additive, and the processing of the wastes to make a felt does not affect them. Processing of the materials on the other hand may decrease the sulfate content. This is due to the fact that the greater part of the hull and seed mate-

[4] Hawk, P. B. and Bergeim, O.: Practical Physiological Chemistry, P. Blakiston's Sons, Philadelphia, 10th Edition, 1931, p. 708.

[5] Mosher, H. H.: J. Biol. Chem., *99*, 781 (1933).

[6] Van Slyke, D. D. and Cullen, G. E.: J. Boil. Chem., *24*, 117 (1916).

[7] Van Slyke, D. D. and Zacharias, G.: J. Biol. Chem., *19*, 181 (1914).

*The distilling apparatus is a modification of the Parnas-Wagner micro-Kjeldahl distilling apparatus[8]. Replacing the distilling bulb is a stopper fitted with the steam inlet tube and a small trap connected to the condenser. The stopper is of such size that it will fit tightly in the large test tubes used.

[8] Pregl., F.: Quantitative Organic Microanalysis, P. Blakiston's Sons, Philadelphia, 2nd English Edition, 1930, p. 111.

RESULTS

TABLE I—NEW COTTON WASTES

No.	Type	Fluorescence	% NH_3	% Urea	% Na_2SO_4
1.	Indian raw cotton	Blue	0.001	0.005	0.23
2.	Indian comber waste	Pale blue	.001	.007	.11
3.	Indian strippings	Pale blue	.001	.007	.07
4.	Indian superior fly	Yellow ivory	.003	.000	.12
5.	Indian ordinary fly	Yellow and brownish blue	.002	.007	.16
6.	Indian low fly	Light tan	.005	.007	.30
7.	Indian superior droppings	Blue	.005	.006	.32
8.	Indian medium droppings	Pale blue	.012	.006	.18
9.	Indian low droppings	Yellow	.016	.007	.51
10.	Indian extra low droppings	Yellow	.023	.003	.36
11.	Indian colored fly	Vari-colored	.001	.004	.11
12.	Indian colored picker	Vari-colored	.005	.002	.24
13.	Indian low colored waste	Vari-colored	.005	.002	.21
14.	Indian sweepings	Blue	.004	.009	.43
15.	Jap. superior picker	Blue	.014	.008	.53
16.	Jap. superior fly	Pale blue	.011	.007	.43
17.	Jap. low fly	Gray-blue	.015	.006	.79
18.	Jap. bleached waste	Pale blue	.004	.002	.09
19.	Jap. sweepings	Blue	.014	.007	.64
20.	Amer. crushed cotton seeds covered with lint	Purple	.017	.005	.10
21.	Amer. comber waste	Pale blue	.000	.003	.15
22.	Amer. strippings	Pale blue	.000	.002	.19
23.	Amer. clean fly	Blue	.008	.003	.31
24.	Amer. dirty fly	Blue	.006	.003	.47
25.	Amer. clean picker	Blue	.007	.006	.26
26.	Amer. dirty picker	Blue	.011	.006	.50
27.	Amer. sweepings	Blue	.005	.007	.24
28.	Amer. colored napper	Vari-colored	.000	.002	.12
29.	China raw cotton	Pale blue	.002	.006	.15
30.	China strippings	Pale blue	.015	.006	.51
31.	China high picker	Brownish blue	.022	.008	.47
32.	China low picker	Brownish blue	.008	.005	.25
33.	China droppings	Gray-blue	.011	.006	.72
34.	China superior fly	Brownish blue	.004	.006	.45
35.	China medium fly	Gray-blue	.000	.004	.53
36.	China low fly	Brownish blue	.003	.004	.58
37.	China choice waste	Brownish blue	.008	.005	.49
38.	China fancy waste	Brownish blue	.005	.005	.38

TABLE II—SECOND HAND COTTON FILLINGS

	Type	Fluorescence	% NH_3	% Urea	% Na_2SO_4
A.	Linters	Purple with spots of ivory	0.063	0.002	0.66
B.	Felt	Yellow	.022	.002	.39
C.	Repicked felt	Purple and ivory	.022	.019	.33
D.	Felt	Ivory	.016	.003	.22
E.	Linters	Ivory	.043	.001	.77
F.	Felt	Ivory	.117	.004	1.96
G.	Felt	Ivory	.162	.006	1.21
H.	Linters	Pale tan	.078	.005	0.75
I.	Felt—50% new and 50% used for 5 years	Ivory with slight bluish case	.023	.052	.29
J.	Linters	Tan	.024	.003	.42
K.	Linters	Ivory	.045	.009	.43
L.	Linters	Ivory	.026	.017	.57
M.	Felt	Ivory	.051	.054	.53
N.	Felt	Ivory	.018	.013	.38
O.	Linters	Ivory and blue	.063	.006	.53
P.	Felt	Tan	.070	.077	.66
Q.	Linters	Ivory and blue	.034	.011	.52
R.	Felt	Ivory	.032	.048	.40
S.	Felt	Ivory	.024	.025	.42
T.	Linters	Ivory and tan	.046	.004	.55

rial is removed during the process preliminary to felting the fibers. Hull material has a higher sulfate content than cotton fibers. Therefore, ammonia and urea contents will not be affected by the processing of the wastes in felt manufacture.

Urease, the urea splitting enzyme, used to determine urea, has been shown to be present in seeds of numerous plants[9]. The presence of the small amount of urea in the new waste materials in somewhat constant concentration, indicates that urea may be present because of the presence of the urease. Enzymes are reaction catalysts, and as such will tend to reverse a reaction under proper conditions. The usual reaction of the enzyme to hydrolyze urea to ammonium carbonate can be reversed to form urea from ammonia, carbon dioxide, and water. To show the presence of urease, a sample of dirty picker waste containing much seed and hull material, was digested in cool distilled water, and to it was added a dilute solution of urea of known concentration. A control was run simultaneously. The sample of waste was analyze for ammonia and urea. The cotton waste was kept in

[9] Takeuchi, T.: J. Coll. Agric., Tokyo, *1*, 1414 (1909).

TABLE III

Felt	*Type*	*Fluorescence*	% NH_3	% *Urea*	% Na_2SO_4
No. 1	SIP—India picker	Spotted yellow and blue	0.016	0.006	0.57
	SIF—India fly	Blue	.002	.005	.45
	Felt—No. 1	Dull pale blue	.004	.005	.34
No. 2	KIP—India picker	Blue	.011	.003	.65
	KDF—Domestic fly	Blue	.001	.006	.27
	KCF—China fly	Pale blue	.012	.006	.69
	Felt—No. 2	Dull pale blue	.010	.005	.47

the urea solution for 18 hours. After that period, the solution was filtered from the waste and the filtrate analyzed for urea. The amount of urea lost by enzyme activity in the cotton waste amounted to 28% of the urea present, and this appeared as a corresponding increase in the amount of ammonia. The presence of urease in cotton wastes explains the presence of approximately the same small amount of urea in new waste samples that have not been subjected to body contamination, and also explains why in some of the second-hand samples of Table II the urea was low and ammonia high.

SUMMARY

1—A method has been developed for the determination of ammonia and urea in cotton materials.

2—Unused cotton materials have both an ammonia and urea content of less than 0.030% and 0.010% respectively. Used cotton fillings will have in most cases either an ammonia content above 0.030% or a urea content above 0.010% or both.

3—Garnetting cotton materials into felt does not alter the ammonia and urea contents.

Roll Treatment—

(Continued from page 452)

steaming of the piece. One salient point with regard to the decatizing operation is often lost sight of and that is the fabric, whatever it may be, is not directly exposed to the steam and therefore the effects are not as great at any aime as the steaming process in the preparing department.

In steaming after crabbing the pieces are not sheltered as in decatizing by means of a thick blowing wrapper which protects the piece to a remarkable degree. Even with a pressure as great as 351 lbs. per sq. inch, a pressure which is often used in decatizing, there is seldom evidence of any wool fibers being burnt or of any browning of the piece goods. To take a case in point, many finishers decatize goods, in the grey state, which they consider are too delicate or are otherwise unsuitable for crabbing. It will be found that in any goods that have been decatized as a preparatory setting process to scouring that the goods can be made to cockle by the application of even moderately warm water. This can never be accomplished with those goods that are steamed upon the crabbing machine for the setting of the fabric here is permanent and cannot be disturbed even when the goods are subjected to the dyeing process which always has a much higher temperature than the scouring operation. This alone shows beyond doubt that the effects upon the cloth as regards steam, tension and pressure are never as severe as in the steaming operation in the preparing department.

This brings us to the fact that only in isolated cases can any damage be caused to the wool fibers of the fabric during the decatizing operation in finishing. The faults here are limited to those faults that occur due to the pressure and tension placed upon the goods such as watering or embossing, cross-twilling of the fabric, etc. The tension and pressure placed upon the goods are determined by two means during this operation: 1. by the tension rails of the machine when the goods are being run on; 2. the tension supplied by the brake drums which command the tension placed upon the blowing wrapper. Both of these require attention if the foregoing and other faults are to be avoided during the decatizing of the goods.

Much is heard nowadays of large diameter can machines and the benefits that can be gained etc., both with regard to handle and finish and production. The writer for many years ran one of these machines with a double length wrapper, the wrapper being run from one steaming can over a drying can at the top of the framework of the machine directly to the steaming can at the opposite side of the machine. This enabled two pieces at one side to be run on and steamed together. At the same time as the steam was turned on for the steaming of the pieces two pieces were commenced to be run in at the opposite side; thus the process was carried out incessantly. It should be remembered that by this method the second piece to be run on went on much quicker than the first and the first piece to be run off came off much quicker than the second one owing to the greatly increased size of the roll. This led to a great increase in production without upsetting the employees, without causing them to work any faster, without speeding up of the machine or without any deterioration in the standard of work turned out by the concern. Formerly one of these machines, decatized on an average, no cooling being attempted and all pieces being run off the machine at steam heat, about eight pieces per hour, this being roughly 500 yards per hour, per machine, per two men. With the new wrapper arrangement fifteen pieces were decatized, or roughly 900 yards. After a little time the machine was averaging eighteen pieces per hour without any great effort on the part of the employees.

This is a typical example of what may be done with the roll treatment of pieces and it is a subject that has not yet been properly explored or understood by the majority of textile dyeing and finishing concerns. Pressure and tension are two of the most important factors in the textile industry and as such cannot be disregarded lightly.

Technical Notes from Foreign Sources

Matte Artificial Silks, with Special Reference to Print

W. Taussig—Monats. f. Seide u. Kunst-Seide 38, 295 (1933).—A two-page article, to be continued; of a practical nature.

Beginning with the first artificial fibers, the strong, almost violent luster which they possess has been, first, their chief appeal, as so many times more lustrous than natural silk; and, next, a point of attack. From almost the first, the characteristic high luster of the artificial fibers has been utilized for some effects, and has also been the subject of numerous successful efforts to deaden it down, at first only to the same luster as that of natural silk. The genius of the tendency has naturally led chemists still further along the same path, even to the devising of methods which deprive the lustrous fiber of all luster whatever; in which case, for some weaves, one might as well have avoided the trouble of preparing the artificial fiber and then delustering, and used ordinary cotton directly. Such efforts seem to the author to be overstrained; this matting down to the luster of natural silk he calls Edelmattierung, but the total destruction of all luster he calls the production of a "verunedelten Ware." We have no exact equivalent in English for the adjective "edel," as used in textile work; its radical sense is "noble"; but the author's phrases may be taken as "ennoblement" and "absolutely degraded goods."

He divides the production of matte effects into two sections: delustering during spinning, and delustering of the finished fiber, either as delustering in the flat (weaves) or by local action by printing upon weaves.

The first method, delustering during spinning, falls within the field of fiber manufacture. It is carried out on an increasingly large scale, and depends mostly upon the incorporation or production of very finely divided substances within the fiber. The method has the advantage of absolute stability of result and uniformity of effect, and these advantages are being recognized more and more by the industry. Details of methods are given, among other references, in Seide, 1929, p. 173; Kunst-Seide, 1929, p. 279, and 1930, p. 102; and Seide, 1931, p. 409.

The second method, that of delustering after spinning, belongs to the field of the finisher. The methods available depend upon the nature of the fiber, whether acetate or viscose. Acetate silk can easily be influenced by various chemical methods, and some physical agencies also produce a diminution of luster, without saponification of the substance taking place. References for this group of methods may include Seide, 1931, p. 399, 447, id., 1932, p. 246, id., 1931, p. 324, 358, 402, 439, id., 1932, p. 193, and Mell. Textil-Ber., 1932, p. 483.

In the case of viscose or cupro silks, it is not possible to bring about a chemical or physical change in the fiber, for purposes of delustering, by any simple treatment. In case of such fibers, delustering, as far as we know at present, is accomplished only by printing on or precipitating a substance insoluble in water (metallic soaps, thin fat or wax emulsions, pigments, etc.)

Definite practical information is given regarding acetate silk. The best-known method for delustering this fiber depends upon the use of a solution of phenol and soap, 2-3 g. of phenol and 5-10 g. of Marseilles soap per liter, the goods being treated in this bath at about 95° C. for an hour, then rinsed at about 75° C., finally with cold water, until the odor of phenol has entirely disappeared. An advantage of the process consists in the fact that the bath can be used continuously, if brought up to strength after each time of use. The phenol should be added in solution to the soap-bath before entering the goods, in order to secure uniformity of content throughout the bath; and any addition even of phenol solution during the working must be avoided, as it is likely to produce uneven delustering. Technically pure phenol (free from cresols) is used. The process works well; but uneven results are obtained if local variations of temperature are allowed to occur in the bath; so that most workers take care that further application of heat (to keep the temperature up after entering the goods) be made at a reasonable distance from the nearer surface of the goods.

Another good procedure consists in padding the goods in a bath of a proprietary compound, Opalogen A (I.G.), containing also some glycollic or lactic acid; then steaming for a short time, and rinsing. A specimen bath consists of Opalogen A, 250 g., lactic acid of 50%, 50 g., the two dissolved in water and made up to a liter. After padding and drying, the goods are steamed for 10-20 minutes, then washed. It is better not to steam in one of the modern steamers (as the Mather-Platt), since any folding or creasing "burns into" the weave, and is next to impossible to get out again. The delustering is very clean and full in effect; it apparently is due to a structural alteration in the fiber.

If the goods, padded as above in the Opalogen A bath, are printed with a paste made up with gum Senegal (merely the paste, without additions necessary), and are steamed and rinsed, a luster-effect is produced on the printed areas—an interesting and puzzling fact. It is difficult to explain, when one considers that, as a development of the delustering method just described, if the naturally lustrous weave be printed with a paste containing the above proportions of Opalogen A and lactic acid, and then steamed and rinsed, a matte pattern is produced upon the lustrous ground. Apparently the phenomenon is dependent upon the particular thickening agent employed, since this peculiar effect is obtained with gum Senegal or British gum, while with gum tragacanth the effect is much less evident.

It will be seen that the above makes possible certain desirable effects upon weaves—colored lustrous patterns upon a matte ground, or colored matte patterns upon a lustrous ground. For the first, a suitable dyestuff is added to the printing paste, and printed upon the goods padded with Opalogen A, and the goods then steamed. For the second effect, the dyestuff is added to a paste containing Opalogen A, and this is printed upon the weave, which is then steamed and rinsed. Among the dyestuffs suitable for the second method are those specifically recommended for acetate silk, also the Indigosols, and even basic dyestuffs. If the Indigosols are used, they are dissolved together with Sol Developer GA and D, and the mixture added to an Opalogen A paste which is prepared and ready for use. After steaming, the goods are treated in a weak nitrite-sulfuric bath as usual, to insure complete development of the color.

Opalogen A does not saponify the acetate silk, so that a weave containing fully or locally delustered fiber can be over-dyed with an acetate silk dyestuff; and, further, by varying the amount of Opalogen A, different degrees of delustering can be brought about, thus adding still further to the list of possible effects.

In dyeing delustered acetate silk, a greater amount of dyestuff is needed in order to attain the same depth of tone as would be gotten with the lustrous form of fiber. It is, e.g., impossible to dye delustered acetate silk to a full black, which can be done with the lustrous fiber; so that it is necessary to dye the black, and to deluster with phenol, in the same bath concurrently.

Indanthrene dyestuffs, when dyed upon acetate silk, necessarily produce a slight loss in luster, though this can be so controlled, according to circumstances, that it is no detriment to quality. Not all of the Indanthrenes, however, give good results (a list of those which the author recommends is given in the paper). The choice of the thickener for the paste is also of importance—it is best a mixture of gum Senegal and dark-burnt starch (brown dextrine?).

Tris-azo Dyestuffs (Mordant)

German Patent No. 477,061 (*June* 3, 1929)—*I. G. Farben-Ind. A.-G.* — 5-Amino-salicylic acid or amino-phthalic acid, or derivatives of either, are diazotized and coupled to an amine capable of being coupled to in para position. This amino-azo dyestuff is diazotized and coupled to another molecule of the same amine or to one of like coupling properties, with the further condition that one at least of these two amines contain at least one sulfonic group. This disazo dyestuff is diazotized and coupled to salicylic acid or a derivative or a homologue of it. Also, any other method may be chosen, which will produce a dyestuff of the type A—N=N—B—N=N—C—N=N—D, in which A is the radical of salicylic or phthalic acid, or a derivative or homologue of either. B and C being aromatic radicals one at least of which must contain at least one sulfonic group, and in which the azo linkages are in the para position to each other, while D is the radical of salicylic acid, or a derivative or homologue of it.

The products (one example is given) are yellow-brown to dark-brown dyestuffs, which are recommended to be converted to chromuim derivatives, by prechroming in substance as usual (heating with chromium formate, with or without pressure), or by afterchroming their dyeings. The tones are about the same by either method of chroming; the dyeings are very fast to boiling. Cotton can be dyed with the products in hot or cold bath. Coppering gives the same results as chroming.

A New Procedure for Printing Upon Silk with Chrome Dyestuffs

"H." — *Monats. f. Seide u. Kunst-Seide* — 39, 340 (1934).—A short, practical article, based upon a recent German Patent, No. 583, 204, held by Durand-Huguenin A.-G. of Basel.

Little use has been made of possible methods for printing chrome colors upon natural silk so far, though the process is entirely feasible. The reason for the only slight or occasional use of such printings lies in the fact that gum pastes, otherwise so suitable for silk, cannot be employed, for, with chromium salts, e.g., the acetate, coagulation of the gum takes place in steaming, and the printed areas become hard and stiff; and subsequent rinsing or soaping of the goods does not alter the stiffness of these printed parts; furthermore, the printed areas will have lost the luster of the unprinted silk, at least partly, or even wholly. When the process is used, the paste has to be made up with burnt starch or detrine, which do not give rise to the difficulty just mentioned, but which have their own disadvantages, of giving less full dyeings, and less sharply defined imprints, than do gum pastes. If the chrome dyestuffs are printed upon silk with a gum paste, but without chromium salts, the prints are satisfactory, and are not hard in texture. But to print in this manner is to relinquish the much greater fastness given by chroming, as well as the characteristic tones perhaps given by mordanting.

The process described in the patent gives prints upon silk with chrome dyestuffs in a gum paste containing chromium salts, without the printed areas becoming hardened, or the luster being diminished, by the inclusion in the paste of a soluble sulfocyanide, an aliphatic polyalcohol, and a substance of the urea group, as ure, thiourea, guanidine, etc. The alcohol may be glycerol, glycol, ethyl-glycol, acetyl-glycol, etc. The prints are deep in tone, and bright, and the scroop is not altered; of course, the fastness is much greater because of the dyeings being chromed.

The dyestuff is pasted up with water, and, say, glycerol, urea, and ammonium sulfocyanide. The thickener (gum paste) is then added, and the mixture warmed upon the water bath to complete solution. After cooling, the chrome mordant is added. The paste thus prepared is printed upon the silk, the goods dried, steamed for one hour under light pressure (0.2 to 0.3 atm. over-pressure),

rinsed in cold water, and finally washed carefully in water at about 40° C. The final wash-water should be almost colorless.

If the dyestuffs are such as to require organic acids in their application (formic or acetic), as Chromacetine Blue S, Modern Blue, Modern Violet, Modern Heliotrope, etc., they are first pasted up with the acid, possibly with addition of hydrosulfite solution, the glycerol, urea, ammonium sulfocyanide, and the gum paste being then added. The rest of the treatment is as before. The ammonium sulfocyanide may be replaced by the potassium, sodium, or calcium salts if desired, and the alcohol may be almost any of the modern glycols or "glycerols" or their derivatives.

A table is appended, giving exact recipes for carrying out the process.

Anthraquinone-azine Dyestuffs

German Patent No. 498,292 (*May* 21, 1930)—*I. G. Farben-Ind. A.-G.*—The process here protected really consists in the purification of "N-dihydro-1-2-2'-1'-anthraquinone-azine" as previously known. The technical product is dissolved in conc. sulfuric acid, and water carefully added until the concentration of the acid is about 15%. To this acid solution is rapidly added a paste of manganese dioxide (manganese mud, not ground pyrolusite) in 85% sulfuric acid (this paste having been prepared below 45° C.), and run in at a temperature which must not rise above 50° C. The whole mixture is then warmed to 70°-75° C., and stirred at that temperature for some hours. The red-brown to orange suspension is then run into water, sodium bisulfite solution added, and the mixture heated to boiling for one-half hour, the red-orange color of the mixture turning to blue. The mixture is then filtered, the solid washed till neutral, and kept as a paste. It is more fast to chlorine than the crude product (apparently the impurities there present have given the dyestuff its reputation in this particular), and the dyeings are purer in tone.

In the process as described, the azine is oxidized, and the impurities destroyed. The bisulfite treatment then reduces the oxidized product back to the original azine.

● PHOTOELECTRIC COLORIMETER

American Instrument Co. announces the production of the new Yoe Photoelectric Colorimeter which was developed by John H. Yoe of the University of Virginia. It is stated that this instrument has had several years of laboratory use and that in addition to relieving eyestrain and increasing the ease of making comparisons it is also capable of giving quantitative results more accurately than any method of visual comparison. Samples of either 5 ml. or 100 ml. may be used and quantitative readings are given directly, referred either to a standard solution or to a Corning filter.

Open Forum

(*See previous issues for details of this department*)

28.—I am in need of some information in regard to the dyeing of cotton ducks with vat dyes.

The method I am using is the regular pigment pad method, reduction being carried out subsequently in jigs.

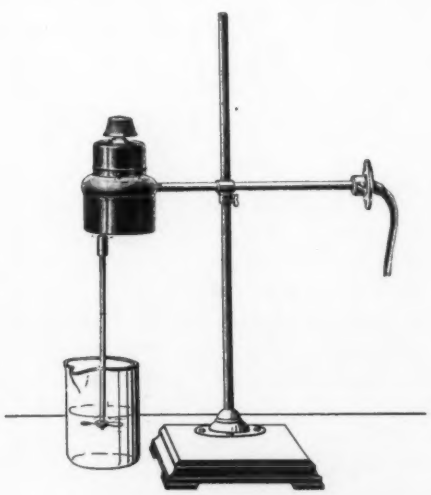
In padding 8 oz. and 10 oz. army ducks what density top and bottom padder rolls would be best adapted? Our pad rolls at present contemplated are bottom, 20 plastometer, top 70 plastometer. We have found that equal density rolls give a shading from face to face. The face next to bottom roll being much darker under this condition.

Should this fabric pass under an immersion roll in the pad box before entering the nip, or, will the method use on light weight goods, passing the cloth directly into the nip having the bottom padder roll partially immersed in the pad box and a spray pipe on the top surface to feed the dye liquor, be best adapted to this type of fabric?

Information in regard to the above will be highly appreciated.—**A. M. L.**

● "LAB-MIX"

A laboratory mixer, known as the "Lab-Mix", has been placed on the market by the Chemical Publishing Co. It is stated that this mixer will be useful in making emulsions, dissolving dyes, gums and resins, etc. A shaded



pole type motor is used which may run for 24 hours daily without damage. It has a free speed of 2800 r.p.m. although the speed may be varied as needed by a rheostat. The shaft and propeller are furnished in either Monel metal or chromium.

● DIRECTOR OF RESEARCH

J. M. Hopwood, president, Hagan Corporation, Pittsburgh, Pa., has announced the appointment of Everett P. Partridge as director of research of Hall Laboratories, Inc. In this position, which he will assume on September 1, Dr. Partridge will be associated with Ralph E. Hall, the managing director of Hall Laboratories and his staff, with the scientific and technical specialists of Hagan Corporation, the Buromin Company, and Calgon, Inc., all of which are allied organizations, and also with the incumbents of the Industrial Fellowship on Calgonizing sustained by Calgon, Inc., at Mellon Institute of Industrial Research.

As director of research of Hall Laboratories, Dr. Partridge will continue his interest in boiler-water treatment and in the processing of water for general industrial use.

● TEXTILE GRADUATES

T. R. Hart of the North Carolina State College has recently completed a survey which indicates that the textile industry is a fertile field for technically trained young men. He has found that during the depression period of the last five years many young men throughout America, upon graduation from college have found it difficult to secure employment, but the textile industry has continued not only to absorb, but to offer real opportunities to young men who were thoroughly trained for service in this field of endeavor.

Professor Hart found that of the 146 students who graduated from the Textile School of North Carolina State College during this five year period, 1931-1935 inclusive, 132, or more than 90%, are gainfully employed in some phase of the textile industry and are making progress.

● DU PONT DYESTUFFS

The Dyestuffs Division of E. I. du Pont de Nemours & Co. has announced the addition of the following products to its line of colors:

Pontamine Fast Orange 6R yields reddish shades of orange on cotton and rayon. Its chief use is as a speck dye and for union dyeing because it is said to dye cotton very much heavier than wool. When dyed by suitable methods, wool is said to be left practically clean. While being recommended for use as a self color, it is said that this new offering may also be employed in combination with suitable colors for the production of speck dye browns.

A new addition to the group of Sulfur Blues is *Sulfogene Brilliant Blue 5G*, a green, bright type. It is claimed that because of its excellent solubility, slow rate of exhaustion, normal rate of oxidation, and generally good fastness, it is desirable for application in closed-type circulating machines, insuring level and uniform results. Because of its brilliance, it is said that Sulfogene Brilliant Blue 5G will be used chiefly as a self color, although it is also suitable as a component in various shades of blues, for application on raw stock and yarns, for chambrays and ginghams.

"Acele" Orange 3R Powder and *Acele Red CR Powder* are said to be suitable for application to acetate fiber yarns and piece-goods, the former yielding bright reddish shades of orange and the latter, medium shades of red. These two colors are said to be particularly useful in the production of dark browns in combination with Celanthrene Brilliant Blue Double Powder, producing economical shades of satisfactory fastness. They are both said to possess good tinctorial strength and to be sufficiently fast to make them perfectly satisfactory for the dyeing of acetate fiber dress materials.

Sulfogene Carbon 2BCF Grains (Patent Applied For)—A new addition to the division's line of sulfur black grain types which produces bluish shades of black on cotton and rayon. It is said to be very soluble and non-dusting, dispersing readily and wetting-out easily, so that no difficulty is encountered from floating on the surface. Furthermore, because of its physical form, it is said to be stable and can be stored for a reasonable period of time without deterioration. It is stated that because of its good application and fastness properties, Sulfogene Carbon 2BCF Grains should find extensive use on all types of inexpensive cotton goods, from drills and jeans to warps for pile fabrics and cotton hosiery. Because of its minimal copper content, it is said that it may also be used on cotton materials to be rubberized.

CLASSIFIED

The rate for "Position Wanted" advertisements in this column is 2 cents a word—with a minimum of 50 cents per insertion. For all other types of advertisements—i.e., help wanted, machinery or supplies for sale—the rate is $5.00 per column inch or less per insertion.

Textile Chemist, B.T.C. degree, Lowell Textile, recent graduate, age 26, single, at present employed as chief textile chemist for cotton manufacturer, but desires change to a concern either textile chemical, dyeing or consulting firm with prospects of future growth and advancement. Qualified for any analytical, research, dye testing, color matching, money values, etc. Excellent command of German. Salary secondary to position with future. Willing to go anywhere. Write Box No. 865, American Dyestuff Reporter, 440 Fourth Ave., New York, N. Y.

OPPORTUNITY—Established organization importing and manufacturing gums, glues, etc., with headquarters in the East wants representation in the South by an individual or company with established contacts, able to handle the products as a side line. Write Box No. 866, American Dyestuff Reporter, 440 Fourth Ave., New York, N. Y.